CHAPTER 2

LITERATURE SURVEY

2.1 Introduction

Wrought nickel based superalloy EI 698 VD is mainly used in compressor parts of turbines. J. Zrnik [7] studied about the effects of structure modification by heat treatments on creep properties. He had found that the optimal creep properties obtained from creep tests and proper microstructure for this alloy were carried out using a heat treatment condition as follows:

1. Solution treatment at 1,100°C for 8 hrs. and subsequent air cooling

2. Precipitation treatment at 1,000°C for 4 hrs. and subsequent air cooling.

3. Aging 775°C for 16 hrs. and subsequent air cooling.

The size of gamma prime particles and the extent of fine carbide precipitation and size on grain boundaries depend on annealing temperatures in individual stages. This heat treatment condition is also used commercially to produce substructure with having bimodal distribution of spheroidal gamma prime pricipitates with an average particle size of 50 nm.

In case of constant loading creep tests at stress levels 663 and 728 MPa at temperature of 650°C, the alloy gave life time 259 and 106 hrs., respectively. These satified agreement in TU-73 standard of aviation that in creep test, the alloy must have ability to resist the creep stress level at 706 MPa at temperature of 650°C for at least

50 hrs. It was also reported that in the fracture specimens showing three rupture modes. Such as intercrystalline brittle fracture, transcrystalline brittle fracture and transcrystalline ductile fracture were revealed. A substructure on fracture surface showed narrow stacking faults inside the larger gamma prime particles. Slip bands also occured close to fracture surface. It was found in EI 698 VD [7] that both dislocation shearing and Orowan bowing interaction with gamma prime precipitates on the creep rupture tests at elevated temperature of 650°C in the range of stresses 580-730 MPa.

The material in this investigation was the polycrystalline wrought nickel base superalloy (EI 698 VD). The chemical composition of this alloy, as shown in Table 1, was quite similar to Inconel wrought alloy grade X-750.

Element	C	Cr	Al	Ti	Mo	Nb	Fe	Si	Mn	S	P	Ni
wt.%	0.08	13- 16	1.3- 1.7	2.3- 2.7	2.8- 3.2	1.8- 2.2	2.0 max.	0.6 max.	0.4 max.	.007 max.	.015 max.	bal.

Table. 1 The Chemical composition of EI 698 VD alloy

Three steps of heat treatment were applied to achieve an optimal microstructure of the alloy which must satify the aviation testing standard TU-73 [7]. The alloy which passed this heat treatment condition has mechanical properties at ambient temperature as follows:

Rupture strength (R_m) $\geq$ 1127,7 MPa.

Yield strength $(R_{p0,2})$ $\geq$ 706 MPa.

Extension percent (A_5) $\geq$ 17 %

Reduction area percent (Z_5) $\geq$ 19 %

The initial microstructure characteristic of the alloy will be investigated by optical microscope. These investigations will include the grain size, morphology and distribution of coarse carbides particles, and its distribution as well. Grain boundary carbide precipitation characteristics will be analyzed using TEM

2.2 Properties of Superalloys [1].

2.2.1 Mechanical properties

The prime reason for using superalloy in gas turbine engines is its high strength at elevated temperature. The microstructure type of nikel base superalloys are close-packed face centered-cubic (FCC) lattice of austenitic compositions. They have good tensile, good rupture, good creep properties, excellent modulus and diffusivity of the FCC lattice for secondary elements. The most importance is the both of abilities to solute many elements in the austenitic matrix and control intermetallic compounds precipitation such as γ ' for strength. In addition, strengthening is also obtained by solid solution hardening and carbides precipitation. By these reasons, superalloys are proper for medium and high temperature service for turbine wheels, turbine blades for short-lived military aircrafts, high long-time creep-rupture resistance for industrial turbine and commercial aircraft buckets, and all types of vanes. Furthermore, for design of complex parts in gas turbines, these mechanical properties are considered

which are good ductility, and good resistance for high-cycle and low-cycle fatigue as well as thermal fatigue.

2.2.2 Physical Characteristics [1]

Density. Nickel-base superalloys have a wide range densities. They depend on the composition of other elements such as aluminium and titanium which make a low density and tungsten and tantalum which make high density. Density is very important consideration for design of rotating components to minimize centrifugal stress.

Thermal Expansion. Thermal expansion is important design factor to operate most performance with close tolerances. Low coefficient in superalloys is used for minimizing thermal stresses which prevents buckling and thermal fatigue cracking.

Thermal Conductivity. High thermal conductivity in superalloys is considered to be used in turbine engines for dissipating heat and minimizing temperature gradients, thus reducing thermal stress and will decrease tendency for thermal fatigue failure.

Phase Instability. By the nature of superalloys, the individual phase has tendency to change their formation when expose to high temperature for long time. Thus heat treatments are used for developing the optimum composition, the size, the shape, the amout of carbides and intermetallic phases. When the base metal has too highly alloyed with certain elements. This can weaken or embrittle the structure, but

for nickel is better than other base metals since it has more resistance for alloying additions.

Oxidation. Oxidation is the reaction of an alloy with oxygen, free of contaminant such as sodium, sulfur, and vanadium. Good oxidation resistance is obtained by a tigh continuous surface scale formation which is diffusion barrier. High aluminium content will form the protective oxides Cr_2O_3 and Al_2O_3 , as well as yttrium, lanthanum. or cerium promote scale retention to make good oxidation resistance in superalloys.

Hot Corrosions. Hot corrosion is combination effects between normal oxidation and reaction with sulfide and other contaminants which occur by combustion of mixture air and fuel. Molten Na_2SO_4 dissolves the protective oxide so sulfur can penetrate through oxide layer into base metal which has reaction of sulfur with chromium that make precipitation of internal sulfide. Hot corrosion will not occur at temperature above 980° C, where oxidation will dominate.

2.2.3 The Metallurgical aspects of Nickel-Base Alloys [1]

2.2.3.1 Chemical Composition

Nickel-base superalloys have many composition elements which are nickel, chromium, cobalt, molybdenum, tungsten, tantalum, columbium, aluminium, titanium, iron, manganese, silicon, carbon, boron, zirconium, and other elements.

The elements can be devided in three main groups:

1. Elements which make FCC austenetic matrix, that consist of nickel, cobalt, iron, chromium, molybdenum, tungsten, and vanadium.

2. Elements which make γ ' precipitation of Ni_3Al, that consist of aluminium, titanium, colunbium, and tantalum (titanium, columbium, and tantalum can substitute for aluminium in Ni_3Al).

3. Elements which make a segregation on grain which consisted of magnesium, boron, carbon, and zirconium

2.2.3.2 The Effects of Alloying Elements [1,10]

Nickel It helps to make FCC austenite matrix, and it is a good base metal which can stand for high solid-solution elements addition in alloys without detrimental phase formation. In some elements, there is phase instability which is a result from phase formation that can weaken or easily to brittle when the base metal has elements addition.

Chromium Nickel-base superalloys which contain chromium content, it will make an increase in oxidation and hot corrosion resistance due to chromium will make a protective oxide (Cr_2O_3). In addition, chromium is element that makes a solid solution hardening which cause alloy strengthening. As well as, it can form a carbide at grain boundaries to make grain boundary strengthening.

Aluminium In general, nickel-base superalloys which have composition of high aluminium content will increase oxidation resistance, because it can make a

protective oxide (Al_2O_3). Furthermore, it produced the γ ' precipitation that is FCC coherent intermetallic phase Ni_3Al which results in superalloys have strengthening, rupture strength increasing. However, if there is too much high aluminium content for making oxide film and segregation, it will increase difficulty for hot working in forming.

Titanium   This element will make age hardening by precipitation of γ ' to make alloys strengthening and also increase hot corrosion resistance. However, if there is too much high titanium content for making oxide film and segregation, it will increase difficulty for hot working in forming.

Molybdenum This element will produce solid solution and carbide forming strengthener on grain boundary which make alloys strengthening.

Columbium   It will increase strain-age cracking resistance in welding due to age hardening of columbium has slower yield aging than mixing with aluminium and titanium (alloys which have composition of columbium will use in temperature that under 705° C). In addition, columbium will form with nickel to be FCC γ ' forming that will make alloys strengthening.

Iron   This element help to make FCC austenetic matrix. Iron can be used for low temperature service because iron can not stand at high temperature. However, iron can be substitute by refractory metals such as vanadium, tungsten, titanium, and etc. However, by this way it will make density increasing in alloys which results for centrifugal stress increasing.

Carbon   Carbon is a composition of carbide by carbon will form carbide with chromium, molybdenum, tungsten, vanadium, columbium, tantalum, and titanium. The common classes of carbide are MC, $M_{23}C_6$, Cr_7C_3, and M_6C.

Tungsten   This element is added to produce solid-solution hardening that will provide alloys strengthening. It also increases density of alloys that is very importance for rotating components design which want to reduce centrifugal stress at minimum.

Tantalum   Tantalum will form with nickel to be FCC γ ' forming that will make alloys strengthening and also increase alloys density.

Cobalt   Cobalt will increase γ ' solvus temperature and improve temperature capability.

Vanadium   This element helps to make FCC austenetic matrix occuring and carbide formation that make alloys strengtening.

Yttrium, Lanthanum, and *Cerium*   They will help to make scale retention and increase oxidation resistance of alloys.

Silicon, Sulfur, and *Phospherous*   They will make a very harmful for alloys. When they are high content, they will make a brittle fracture. However, they have beneficial effect for casting process.

Boron, Manganese, and *Zirconium*   They will make grain boundaries segregation.

2.2.4 Effects of Major Phases on Structure and Microstructure [1]

There are three major phases in superalloys which are following:

2.2.4.1 The Austenite Matrix (γ)

Pure nickel does not have high modulus of elasticity or low diffusivity properties which make high rupture and creep resistance. However, its austenitic matrix structure with other solid solution elements is wanted for using these alloys to $0.8T_M$ (melting point) and for times up to 100,000 hrs. at somewhat lower temperature. The basic reasons for this endurance are given

1. Nickel has a high tolerance for alloying without phase instability.

2. Chromium addition will form Cr_2O_3 , protective scales having low cation vacancy content, thus it can restrict the diffusion rate of metallic element go outside as well as oxygen, nitrogen, sulfur, and other aggressive atmospheric elements go inside.

3. At high temperature, there is Al_2O_3 forming which is protective scales to make good oxidation resistance.

2.2.4.2 Gamma Prime Phases (γ ')

In the γ '-type A_3B compound, there are both electronegative elements such as nickel, cobalt, or iron, compose the "A" and electropositive elements such as

aluminium, titanium, or columbium compose the "B". In nickel-base alloy, γ ' is (Ni, Co)$_3$(Al, Ti) with nickel and aluminium dominating, although adding at least as much titanium as aluminium, for multialloying elements roles.

γ ' by dislocation interaction, it contributes antiphase boundary (APB) strengthening to the γ -γ ' alloy, the strength of γ ' increases as temperature increases. The inherent ductility of γ ' can prevent the severe embrittlement, This is different from strengthening by phases of higher hardness, such as carbides, severe embrittlement can occur by the formation of brittle sigma (σ), and Laves phases.

2.2.4.3 Carbides

Firstly, carbides appeared on grain boundaries as location sites in nickel alloys, thus previous reports told that grain boundary carbides decreased ductility, then many development methods reduced carbon content. However, work from Nimonic 80A and Udimet reported that if there is carbon content less than 0.03%, it would sharply reduce creep life and ductility. Most investigators feel that carbides has beneficial effect on rupture strength at high temperature. In addition, carbide morphology has effect to ductility, chemical stability of the matrix.

2.2.4.3.1 Classes of Carbide and Typical Morphology

The common classes of carbides are MC, $M_{23}C_6$, Cr_7C_3 , and M_6C. MC usually are coarse random cubic or script morphology. $M_{23}C_6$ usually occurs at grain boundaries as irregular discontinuous blocky particles also plates and regular geometric

forms. Cr_7C_3 occurs as a blocky intergranular form. The continuous and/or denuded grain boundary $M_{23}C_6$ and M_6C are avoided for best ductility and rupture life time.

MC carbides. MC carbides form in superalloys shortly below freezing as discrete particles. MC carbides distributed heterogeneously both in intergranular and transgranular position also often interdendritically. The primary carbides are FCC structure, closely packed structure are very strong. They occur by combination of carbon with reactive and refractory metals such as TaC, NbC, TiC, and VC. M atoms can substitute by other less reactive elements such as molybdenum and tungsten which can find (Ti,Mo)C in U-500, M-252, and Rene'77. The change in stability order cited of M atom substitution can weaken the binding force in MC carbides which degenerates reaction to form more stable $M_{23}C_6$ and M_6C type carbides during heat treatments or service. Modern alloy with high columbium and tantalum contents stabilize MC carbides. This will not break down easily during solution treatments.

$M_{23}C_6$ carbides. are ample in alloys with moderate to high chromium content. They form from degeneration of MC carbides and soluble carbon residual in alloy matrix during lower temperature heat treatment and service which temperature is about 760-980° C. They usually occur at grain boundaries but they can also occur along twin line, stacking faults, and at twin ends. $M_{23}C_6$ carbides are a complex cubic structure, which if carbon atoms were removed. Tungsten or molybdenum can also make $M_{23}C_6$ carbide which its composition is $Cr_{21}(Mo,W)_2C_6$. Nickel can substitute in the carbide, cobalt or iron can substitute for chromium as well as other refractory elements can locate in the subscript-2 position. $M_{23}C_6$ carbides which have

critical location at grain boundaries will have effect on rupture strength such as counteract grain boundary sliding. However, at the last, fracture usually initiate at grain boundary $M_{23}C_6$ particles or by decohesion of the $M_{23}C_6$-γ interface.

M_6C carbides. These carbides have a complex cubic structure which form at higher temperature about 815-980° C. They are similar to $M_{23}C_6$ carbides but they will occur when there is high refractory metals content. When alloy contains molybdenum or tungsten more than about 6-8wt.%, M_6C will precipitate at grain boundary, usually with $M_{23}C_6$. Typical formulars for M_6C are $(Ni,Co)_3Mo_3C$, and $(Ni,Co)_2W_4C$. Composition of M_6C can vary very widely. M_6C carbides are stable at higher temperature than $M_{23}C_6$ carbides. M_6C precipitation at grain boundaries can control grain size in wrought alloys.

Cr_7C_3 carbides. They are irregular blocky particles in simple alloys with low chromium contents and low reactive and refractory element contents. When aging, Cr_7C_3 carbides usually transform to $M_{23}C_6$ carbides.

Nickel-base superalloys have high strength by these following structures

1. Alloy matrix (γ). The FCC nickel-base austenitic phase has high solubility for solid solution elements such as cobalt, chromium, molybdenum, and tungsten.

2. Gamma prime (γ'). Aluminium and titanium will form with nickel as Ni_3Al and Ni_3Ti to precipitate for high volume fractions of FCC γ' which has interface bond with austenitic.

3. Carbides. Carbon is added at low percentage about 0.05-0.2 wt.% and react with reactive and refractory elements to form primary MC carbides. During heat treatments and in service, MC carbides will change to be lower carbides such as $M_{23}C_6$ and M_6C along grain boundaries.

4. Grain boundary γ '. Heat treatments will make γ ' film along the grain boundaries which can increase rupture properties.

2.3. Creep

Introduction

Nickel base superalloys are the most widely used as high temperature materials for application in blades and discs of turbines, both in power generation and aeroengines. The service conditions in these machines often combine with aggressive environment, high stresses having time-dependent components with high temperatures. For components like turbine blades the loading can be idealised as stationary over a considerable period of time, and therefore the design engineers generally approximate the response of the component through the application of constant load (or stress) creep data of material [1].

In high temperature, both of constant and uncostant load will make a limit lifetime of metals. This is different from using metals in low temperature. Since in low temperature, at constant load and not more than yield strength point of each metals, lifetime is unlimit. Stress at hight temperature makes specimens have continuous strain, then this result will make creep [16].

2.3.1 Creep Curve [5]

In finding creep curve of metal will use the testing which apply constant load
and constant temperature with tensile specimen, then measure strain which take place
in each time intervals. Then ploting value of strain with time which will produce a
curve as Figure 2.1.
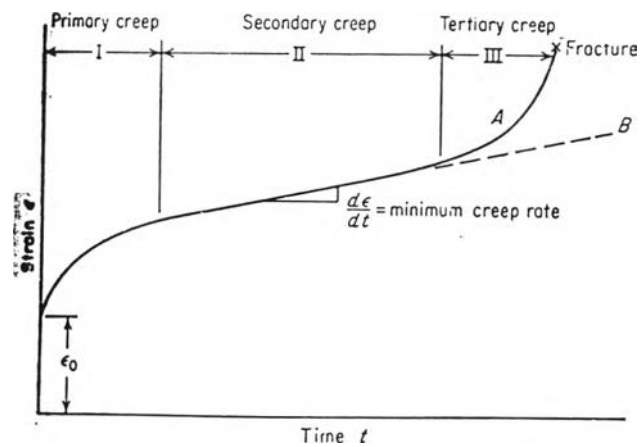
Figure 2.1 Schematic Represenation of High Temperature Creep Curve [5]

From this figure will show that creep curve consists of 3 parts which are

1. Primary creep or transient creep.

2. Secondary creep.

3. Tertiary creep.

All of 3 stages take place after initial strain (ε_0), which occurs immediatly when applying load. And initial strain can occur even stress is lower than yield stress (elastic interval). Even initial strain is not creep but it is important value. Since the value of allowable total strain in parts of machine will include this initial strain, if it has high value. Sometime it must have consideration about thermal expansion.

2.3.1.1 Primary Creep or Transient creep is the interval of creep rate decreasing. This interval has changing in structure which the creep resistant increase by deformation due to work hardening by dislocations piling-up and/or dislocations rearrange to be low angle boundaries (Polygonization). Then dislocations move difficultly, so it makes creep rate decreasing.

2.3.1.2 Secondary creep is a period of constant creep rate which results from a balance between the two competing processes of strain hardening and recovery. Secondary creep is usuallly refered to as steady-state creep. The average value of the creep rate during secondary creep is called the Minimum Creep Rate (MCR).

2.3.1.3 Tertiary creep is interval which has creep rate increasing until to fracture. Tertiary creep occurs when there is an effective reduction in cross-sectional area either because of necking or internal void formation. Third stage creep is often related with metallurgical changing such as recrystallization, over aging or coarsening of precipitate compound particles which will make deformation rapidly. Also including work hardening occuring which reduce flow ability of metals then it will make a fracture later. Sometime environment has effect to creep process such as oxidation

occuring which will reduce cross section area of specimen. This will increase tertiary creep rate. In machine designing for high temperature, data of time and strain before tertiary creep is the most considering.

2.3.1.4 Modified creep

In some conditions, metals do not show all of 3 creep stages. Such as in high temperature and stress, primary creep may not occur and sometime there is only tertiary creep. In contrast, such as in case of cast alloys may not have tertiary creep. Fracture can occur with only little strain. And Figure 2.2 shows the effect of applied stress on the creep curve at constant temperature. It is apparent that a creep curve with three well defined staged will be found for only certain combinations of stress and temperature.
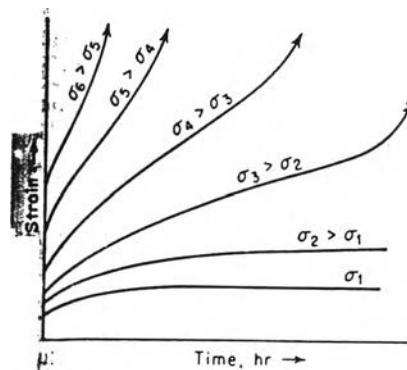
Figure 2.2 Schematic representation of the Effect on Stress Levels on Creep Curves at

Constant Temperature [5]

2.3.2 Creep in two phase Alloys [18]

Major development in creep resistantce can be achieved by a dispersion of fine precipitates or insoluble particles which provide effective obstacles to dislocation movement. Under high temperature creep conditions, dislocation can rarely cut throuugh the particles. Dislocations moving in the alloy matrix must try to rise above the obstacles by climb and cross slip. However, by pinning grain boundaries, particles can also hinder grain boundary sliding and affect the facility of vacancies generating at grain boundaries.

It is essential that the particle dispersion does not coarsen rapidly during exposure service. Particle coasening increases both the average particle size and the mean inter-particle spacing, so the creep resistance will decrease with time as fewer particles are present to hinder dislocation movement.

Precipitate coasening can be determined by reference to A-B alloy, with precipitates consisting of B atoms in a matrix of almost pure A atoms. The concentrations of B atoms in the matrix will increase with decreasing particle radius. The concentration of B atoms in the matrix around large precipitates is lower than around small particles. At high temperature, B atoms will diffuse from the smaller to the larger precipitates, so the larger precipitates can grow. As the small particles disappear with time, the average particle size and the average interparticle spacing increases.

2.3.3 Creep of γ / γ' alloys [10]

In steady state creep of γ / γ ' alloys, there is the presence of a homogeneous network of a/2 <110> dislocations surrounding the γ ' particles and staying for the most part at γ / γ ' interfaces. When increasing the applied stress, which will increase the density of network dislocations strain hardening will increase in steady state occurring by the strong interactions between these network dislocations. Recovery mechanism occurs by dislocation annihilation within networks due to thermally activated cross slip and/or climb processes. Thus, in this steady state interval, dislocations rearrange within the γ / γ ' interfacial networks controlling both the strain hardening and recovery processes. The beneficial effect on creep properties of certain alloying additions, e.g., Co, Mo, and Cr which reduce stacking fault energy of nickel resulting in low stacking fault energy γ matrix phase, which retard the recovery rate of the networks, thus increasing the strain hardening resulted in reducing the steady state creep rate. Also, slow diffusing solid solution hardening elements should retard the recovery rate and decrease the steady state creep rate, W and Re which partition to the γ phase, are potential creep strengtheners of superalloys. Ti, Nb, and Ta partition preperentially to the γ ' phase, will change the anisotropy of the APB energy in γ ' which increases creep strengthening.

2.3.4 Mechanisms of Creep Deformation [18]

1. Dislocation Glide Mechanism, which relates to dislocation moving along slip plane and overcoming barriers by thermal activation. This mechanism will take place at high stress.

2. Dislocation Creep Mechanism, which relates to dislocation movement overcome barriers by thermomechanism such as vacancy diffusion or interstitials.

3. Diffusion Creep Mechanism, which will occur when there is stress helping to produce vacancies and interstitials flowing through a crystal. This mechanism relates to Nabaro-Herring and Coble creep.

4. Grain boundary Sliding Mechanism, which relates to the sliding of grain boundaries in each other.

If there are many mechanisms occur at the same time, they are operating independently of each other. The fastest mechanism will dominate the creep behavior. However, if there are mechanisms operating in series, the slowest mechanism will control the creep deformation.

Creep is a process in which work hardening and recovery processes occur on the same time scale as the deformation process. Several mechanisms of creep depend on temperature and applied stress, dislocation glide, dislocation recovery, (ie., by process involving dislocation climb) , or other diffusional flow mechanisms.

2.3.4.1 Diffusional creep Processes [6]

Diffusion occurs by vacancy movements in crystal lattice. Atom can move to the site of an adjacent vacancy, that for only atom which has enough thermal energy to jump from its original site. Vacancy movement is the dominant diffusion mechanism in most metals and alloys.

2.3.4.1.1 Nabarro-Herring creep [6]

This mechanism involves atomic diffusional flow. Nabarro-Herring creep dominates creep process at much lower stress levels and higher temperature than those at which creep is controlled by dislocation glide. In Figure 2.3, shows Nabarro-Herring creep results from higher temperature and low stress that make a vacancy flux and a mass flux in the opposite direction in grain.
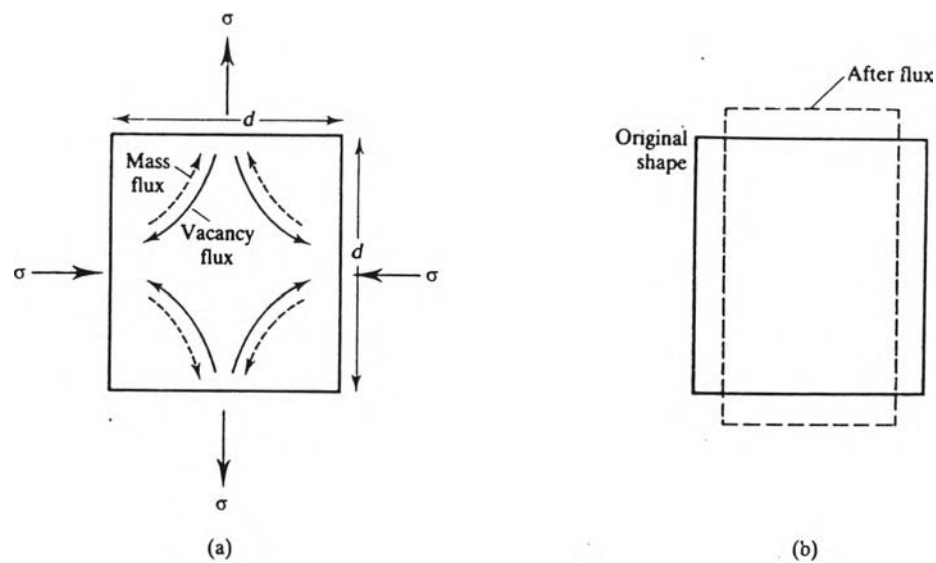
Figure 2.3 Schematic representation of Nabarro-Herring Creep [18]

(a) The resulting change in grain dimensions

(b) is equivalent to a creep strain

Consider a cube-shaped grain in a polycrystal deforming at high temperatures under stresses, vacancies diffuse to the boundaries under compression that resulting in a counter flow atoms. This process is equivalent to move atoms from compression boundaries and plate them on tensile boundaries. By both vacancies and atoms movement at the same time, causes time-dependent extension of the crystal in the tensile direction which will reduced the cross-sectional area of the grain at right angles to the tensile axis.

2.3.4.1.2 Coble creep [6]

Coble creep is same as Nabarro-Herring creep by the vacancy concentration gradient but Coble creep transport occurs by diffusion along grain boundaries in a polycrystal or along the surface of single crystal. Vacancies can transfer from tensile boundaries to those under compression also along the boundaries. When temperature decreases towards about 0.4 T_M, diffusion along preferential paths becomes important as lattice diffusion processes are more difficult. Coble creep will be the dominant process. Coble creep is more sensitive to grain size than Nabarro-Herring creep. Thus Coble creep will dominate in very fine grain materials. For preventing internal voids or crack during diffusional creep of polycrystal, mass transfer process will be used and occur at grain boundaries. These result in grain boundary sliding.

2.3.4.2 Dislocation Creep Processes [6]

Due to the estimated diffusional creep rates are too low by many orders of magnitude, so that alternative theories of deformation are required. The situation is

similar to that of low temperature deformation , theories can predict flow quantitatively only if they allow slip to occur by the passage of crystal dislocations. Slip is observed during creep and electron microscopy confirmed that a change in dislocation density and arrangement occur as creep continues. Very different materials often show similar creep curves, even different crystal structures and dislocations with very different properties, creep curves will still consist of primary and secondary regimes. Any dislocation theory of creep must have general flamework which is provide by the phenomena of work hardening, recovery and internal stress.

2.3.5 Creep Theories [6]

Weertman [6] suggested dislocation climb as the recovery process. During creep, the deforming material which contains of many dislocation sources, will send out dislocations moving across their glide planes until the leading dislocations stop at obstacles and producing a piled-up dislocations, result in an increase of work hardening. The leading dislocations will climb out of its slip plane that they may be annihilated when meeting a dislocation of opposite sign or may be free to glide in a new slip plane after climbing some distance until they meet new obstacles. But at the same time, the source will send out a new dislocation which continues creep process.

In an alternative theory explained that the dislocation within the cell interiors consisting of a three-dimensional network, recovery processes such as climb will make this network coarsening (in a manner similar to grain growth). However, stress will provide the network bowing out and acting as dislocation sources. This multiplication

of dislocations which increases the dislocation density and refines the network size, by these reasons will increase work hardening.

2.3.6 Creep Fracture [6]

The first indication of creep fracture causes by the acceleration in creep rate marking the onset of the tertiary stage. There can be many different causes for this acceleration such as during constant-load test, the stress gradually increases as the cross-sectional area of the specimen decreases with increasing strain which causes the creep rate accelerating. However, even for constant-stress creep test or when changes in specimen cross-section can be regardless with low creep ductilities (1% or so), a tertiary stage normally precedes fracture. In these cases, tertiary may creep by

a) mechanical instability, such as the presence of necking which results in a localized reduction in cross section area,

b) microstructural instability, including grain growth or recrystallization with single phase materials or the gradual loss of creep strength as overaging occurs during creep of precipitation-hardened alloys and/or

c) the nucleation and growth of internal microcracks which develop until the number and size of the microcracks are sufficient to cause the creep rate increasing.

For macroscopic character, it can be both ductile and brittle. If it is ductile, fracture will be transgranular and occurs with elongation and necking. For brittle, fracture will be intergranular which has elongation or necking very little or none of them. In some cases fracture will has both transgranular and intergranular fracture.

firstly intergranular will occur which will reduce cross section area so it will increase stress until it have transgranular fracture at the last. In general transgranular fracture will begin at grain boundaries meeting or will occur at area of one grain boundary meet surface or second phase. In addition, it will have creep cavities at grain boundaries, ecpecially for precipitation phase in grain boundaries. This case, it is no need to occur at meeting point of grain boundaries, This process has been called Cavitation Creep. If there is cavities in grain boundaries which are near cracking, can incicate this fracture cuase by creep.

Type of fracture, beside it depends on temperature, it will depend on strain rate In general, slow creep rate or slow rupture time with high temperature will make intergranular cracking. Furthermore that cracking occurs at grain boundaries, also character of grains are equiaxed, even at high plastic deformation. In case of transgranular fracture, grains will elongate with the same deformation direction

However, it should be emphasized that microcracks development leads to creep fracture. Under high temperature creep conditions, microcracks can form and grow along grain boundaries which can make intergranular fractures which have two forms. Wedge or triple-point cracks are often found at high stresses. For low stress creep, intergranular cracks develop by nucleation, growth and link-up of grain boundary cavities.

Both types of intergranular damage, the voids and microcracks nucleate early in the creep life with numbers and sizes of cavities and cracks increasing as creep

continues, which can make intergranular creep fracture by cavities and/or microcracks linking up to form a crack which can propagate under the applied tensile stress.

2.3.6.1 Triple Point Cracking [6]

Under high temperature creep conditions, the grains within a polycrystalline material can move relative to each other. which produced shearing along grain boundaries. termed grain boundary sliding as shown in Figure 2.4. Thus wedge cracks can form and grow along grain boundary. Sliding on grain boundaries which produces a shear stress can lead to stress concentrations which is high enough to nucleate cracks at triple points and growing by continued sliding along boundaries normal to the tensile stress axis. The relative movement of two adjacent grains in a poly crystalline material also results in deformation of the surrounding grains. Thus sliding can occur which this rate equal only to the rate permitted by grain deformation. The rate of grain boundary sliding or the rate of wedge-crack development, is determined by the overall creep rate.
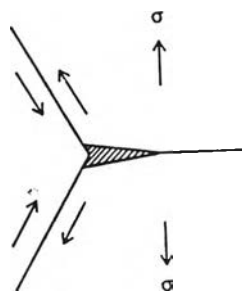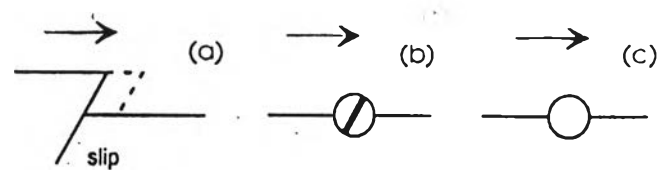

Figure 2.4 Schematic representation of Wedge-Shaped Crack at Triple Grain Junction [6]

2.3.6.2 Creep Cavitation [6]

Nucleation of grain boundary cavities also depends on grain boundary sliding. In this case, large stress concentrations is produced where sliding is held up at small obstacles, which include

a) ledges or steps formed, which is a slip band in one grain intersects the boundary (Figure 2.5a)

b) non-deformable particles or hard inclusions at grain boundaries, with voids forming either by fracturing the particle or by decohering the particle / matrix interface (Figures 2.5b and 2.5c).

Figure 2.5 Schematic representation of Grain Boundary Cavity Nucleation [6]

In another view is that grain boundary sliding is responsible for both nucleation and continued growth of cavities. With the rate of sliding was controlled by the rate of grains deformation, which is described for the dependence of the rupture life on creep rate. However, there are many theories which are based on the concept that, the cavities have reached a critical size, growth can occur by absorbtion of vacancies from surrounding grain boundaries.

2.4 Fatigue

2.4.1 Elavated Temperature Fatigue

In using metals at high temperature, when there is increasing or decreasing of load for sometimes as cyclic load. This will make fatigue in metals. Temperature has high effect to fatigue strength of metals. When temperature increases, fatigue life will decreases.

2.4.2 Fatigue of Superalloys [1]

There are many failures of engineering structure as a result of cyclic loads or fatigue. Fatigue fractures may be classified which depend on fracture path as low temperatures fractures which are normally characterized by transgranular initiation and propagation, and as high temperatures fractures are predominantly intergranular. The transition from low to high temperature behavior is a function of temperature, strain rate, alloy type, and a steady or mean component of stress.

2.4.3 Characteristics of fatigue at low temperatures [1]

The low temperature fatigue fracture process can be devided into four stages

1. Planar slip of heterogeneous nature produces persistant slip bands at free surfaces. These slip bands are normally on slip planes of highest resolved shear stresses, and develop to be microcracks.

2. Linking of microcracks which propagate along slip planes toward the interior of the specimen, leads to formation of a macrocrack.

3. Macrocrack propagates slowly along a noncrystallographic plane which is usually normal to the direction of the maximum principal stress.

4. Crack propagates rapidly, following attainment of the critical length for the material, geometry, and test conditions.

Precipitation hardening can improve fatigue properties under these conditions which are uniform and stable dispersion of a second phase which increase stain hardening capacity and tend to disperse slip. At elevated temperatures for precipitation-strengthened superalloys, the strengthening phases are neither uniform nor stable which reduced fatigue-to-static-strength ratios. In some alloys have tendency for denuded zone formation near grain boundaries and tendency in some alloys for strain-induced solutioning of precipitates, deformation bands develop in these precipitate-free areas and lead to premature microcracking and fatigue failure.

2.4.4 Characteristics of Fatigue at High Temperatures [1]

Fatigue at elevated temperatures shows many characteristics of creep rupture. Grain boundary sliding is important for overall deformation. Fatigue fractures initiate as grain-boundary voids and wedge cracks as in static fracture. At increasing temperatures, crystallographic slip is more homogeneous and slip-band cracking is suppressed in favour of intergranular fracture. In general high temperature fatigue may be thought of as a cyclic creep-rupture process. The combination effects of elevated temperature fatigue and creep damages may be represented by the sum of the plastic strains produced in each.

There is also a transition stage between low and high temperature behavior that fracture initiates intergranularly but propagates primarily transgranularly which results from combination effects, including increased stress intensity and strain rate at the tip of the enlarged crack, and increased resistance to deformation as the crack front moves away from the surface.

2.4.5 The Behavior of Superalloys in Thermal Fatigue [1]

Thermal fatigue is important effect for consideration to very high gas turbine operating temperatures for maximum power and efficiency as well as longer operational lives. The sequence of event consists of the following:

1. A heating cycle during which the component is heated nonuniformly, then giving an increase of internal stresses that are compressive in areas hotter than the instantaneous mean and tensile in areas cooler than the mean.

2. An equilibrium period during which the component operates at a steady state but not necessary at uniform temperature.

3. A cooling cycle during which the component is nonuniformly cooled and during which those areas that were stressed in compression during heating are stressed in tension and *vice versa*.

If there are stresses high enough during the transient heating and cooling cycle, and sufficient repeating number of times, then resulting in localized fracture.

In thermal fatigue fracture are also classified as either low temperature or high temperature, depending on the fracture path which is trangranular on intergranular. The transition from transgranular to intergranular depending on these factors: maximum and minimum cycle temperatures; heating and cooling rates (or strain rate); temperatures at which peak cycle stresses or strains are reached; environment (oxidizing); hold times at steady state stresses, which results a creep damage; and the alloy itself.

2.5 Creep-fatigue Interaction and Cyclic Creep in Structural Alloys [10]

Creep-fatigue interaction relatives to cyclic loading at high temperature which has time-dependent and thermal activating process. A basic measuring of creep-fatigue intereaction are devided in creep and fatigue then combinates their lives. Most deformation at elevated temperature can occur by both creep and fatigue that cause damage materials.

The term cyclic creep, it means a number of testing conditions which have stress controlling with low or very low frequencies. Type of loading conditions have both triangular and square wave loading between zero and tensile stress, or between a tensile and compressive stress, or between two tensile stress. The results of these tests will be compared with static creep tests that will show either cyclic creep acceleration or deceleration. The comparison between the cyclic testing and static creep tests will measure relatively to time on load. Thus if there is no creep-fatigue interaction, the

specimen will fail when the sum of the times on-load is equal to the static creep rupture life.

For load cycling between zero and maximum stress, cyclic creep acceleration which increases minimum strain rate and decreases rupture life, which based on-time on load has been reported for a number of steels. During the off-load period, the hardened microstructure which occurred during the on load period will recover. Thus when there is repeating load, primary creep interval will occur repeatly until work hardening can be observed. These repeating primary creep interval during the initial portion of the on-load make the creep acceleration.

However, it will have cyclic strenthening effect such as reports from previous works [19,20] when it has a load ranging from hours to minitues because there are the storage and recovery of an elastic strain. Minimum strain rate depens on frequency because of anelastic strain storage interacting with nonrecoverable creep. Thus unloading period decelerates the rate of nonrecoverable creep. Both processes, anelastic strain storage and recoverable creep occur during on-load in series. Thus during initial portion of the on load cycle, the storage of anelastic strain occurs and it will delay nonrecoverable creep that will make the cyclic creep deceleration.

From the many behaviors for these particle strengthened system even for the simple square wave loading form, it is difficult to use empirical equations such as life fraction rule to predict lives.