CHAPTER 3

EXPERIMENTAL PROCEDURE

3.1 Introduction

The study of the microstructure, dislocation substructure, fracture mechanism and mechanical properties of wrought nickel base superalloy EI 698 VD as functions of high temperature tests such as tensile, creep, fatigue, isothermal cyclic creep and cyclic creep with thermomechanical fatigue were carried out. The specimens for testing investigation were taken from heat treated as-wrought nickel base superalloy. Mechanical properties were assessed as function of types of high temperature tests.

3.2 Materials

See material's details in literature survey, chapter 2.

3.3. Tensile Tests.

The evaluation of mechanical properties of the alloy was done by longitudinal tensile testing at room temperature and elevated temperature of 650°C at strain rate of 0.28 mm/min. The size and dimensions (in mm unit) of specimens for tensile, creep, fatigue at constant high temperature and cyclic tests at constant temperature are shown in Figure 3.1.

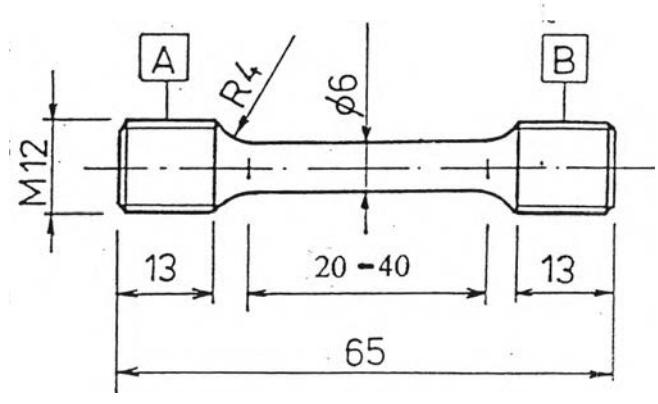


Figure 3.1 Schematic representation of Size and Dimensions of Specimens for Tensile,Creep, Fatigue at High Constant Temperature and Cyclic Creep at Constant High Temperature

3.4 Creep Tests

The creep tests to define the creep strength were carried out at different conditions for evaluating the effects of varying stress levels on life time. The creep tests were carried out on the creep machines VUHZ 2000 at a temperature of 650°C. The stresses were 612, 706, 740, 760, and 850 MPa. All of these creep tests were constant load creep test.

The elongation with time was recorded. The creep deformation was measured by extensometer. The elongation was measured by two micrometers. The average value of these obtained data was brought to plot the creep curves on elongation-time axises. The stress of 740 MPa was chosen as a constant load parameter for evaluation of cyclic creep behaviour of the alloy.

To study the development of creep deformation in dependence on time at chosen creep stress (740 MPa.) and temperature of 650°C, the two additional creep tests were realized. The first test was interupted according to the time period for transcient, the second test was interupted corresponding to secondary creep stage. The creep deformation progress was evaluated by employing TEM of thin foils.

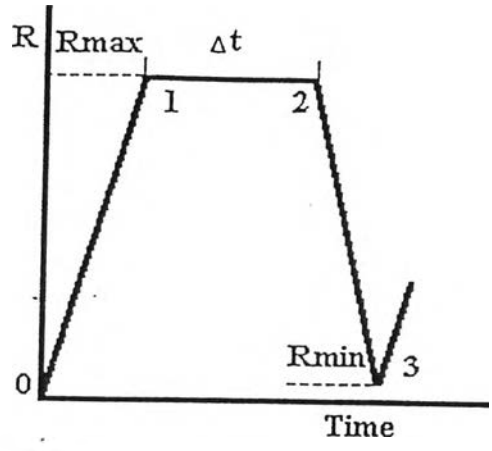
Both additional creep tests were also constant loaded (740 MPa.) creep tests. These tests were started by heating up the specimens until reaching the temperature at 650°C. The temperature for creep was controlled by two Pt-PtRh thermocouples by means of thermal compensator. The temperature was maintained within the range of ± 1°C. The load (740 MPa.) was applied when high temperature was stabilized.

3.5 Cyclic creep tests

3.5.1 Cyclic creep tests at constant temperature

These tests were carried out by the continuous cyclic creep tests with different tension hold times at upper stress load amplitude σ_{max} = 740 MPa. This stress level was selected due to obtained results from creep tests. The wave form of stress as a function of time was trapezoidal shape and was remained unchanged throughout the whole test for each individual hold time as shown in Figure 3.2. Stress was released repeatedly after holding time for 1, 3, 5, and 10 hrs. The remained stress after releasing load was maintained at σ_{min} = 20 MPa to keep the loading system in tension.

Therefore the stress ratio R for each cycle test was R = 0.02703. The testing temperature through out the test was constant at 650°C.



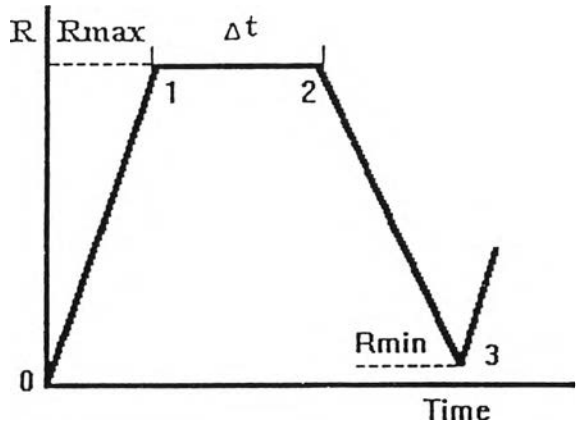
The testing procedure for program A: heating (zero load) → load rising (01) → hold time (12) → load reduction (23).

Figure 3.2 Scematic representation of Form of Stress Waves of Cyclic Creep at Constant High Temperature (650°C)

3.5.2 Cyclic creep tests with thermomechanical fatigue stress component

These tests were carried out by continuous cyclic creep with tension hold time at upper stress amplitude σ_{max} = 740 MPa. The wave form of stress-time was trapezoidal shape and was remain unchanged throughout the whole life time for each hold time as shown in Figure 3.3. Cooling prior to releasing load was done after every holding times at 1, 3, 5, and 10 hrs. in each cycle. The temperature was heated to

650°C by resistant heating. The procedure steps in each cycle was as follows: heating, loading, holding, cooling, load relaxing, then heating and loading again, repeating for each cycle. The air blow cooling was applied to decrease the temperature to 50°C. The stress after load reduction was maintained at σ_{min} = 20 MPa to keep the loading system in tension. The stress ratio R for each cycle test was R = 0.02703. The size and dimensions are shown in Figure 3.4. The specimen is designed to enable localizing the heating and to provide the testing temperature constantly in given gauge length area. The in-situ temperatures at two points on gauge length will be recorded.



The testing procedure for program B: heating (zero load) → load rising (01) → hold time (12) → cooling (prior to load reduction) → load reduction (23).

Figure. 3.3 Schematic representation of Form of Stress Waves of Cyclic Creep with Thermomechanical Fatigue Stress Component

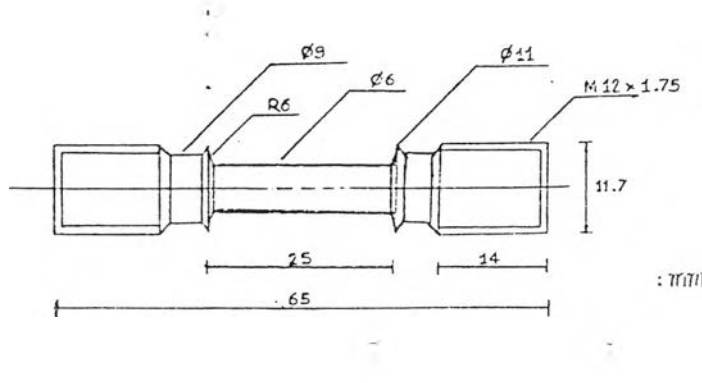


Figure 3.4 Schematic representation of Size and Dimensions of Specimen for Cyclic Creep with Thermomechanical Fatigue Stress Component

3.6 Fatigue Tests

The high temperature (T=650°C) fatigue tests were carried out for comparing with cyclic creep at constant high temperature (T=650°C), in term of holding time equal to zero. The tests were done by using fatigue testing machine having the following conditions as maximum stress was 740 MPa. and minimum stress was 20 MPa. Time for increasing and decreasing stress from maximum stress to minimum stress was 1 min. The fatigue stress wave form is shown in Figure 3.5. The tests were obtained in two tests which were rupture and unrupture fatigue tests. The dislocation substructures from unrupture test was investigated by using TEM.

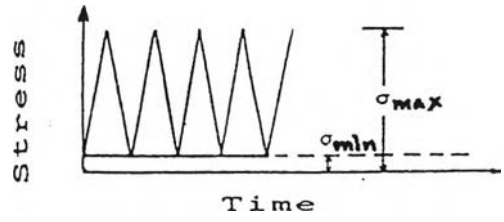


Figure 3.5 Schematic representation of Stress Wave Form of Fatigue Test

The time-dependent deformation in both cyclic creep tests (3.5.1 and 3.5.2) and high temperature fatigue tests (3.6) were measured and recorded by the same method of creep tests (3.4). These tested specimens were also investigated by light metallography. The fracture surfaces for both cyclic creep tests at constant temperature and cyclic creep tests with thermomechanical fatigue stress component, as well as fracture specimen from high temperature fatigue test, were examined using SEM. The fracture mechanism and morphology of fracture were investigated in terms of the effects of holding time, load, load-temperature changes. To evaluate the superimposing of cyclic stress component upon creep stress as a result of cyclic creep at constant high temperature and cyclic creep with thermomechanical fatigue stress component, the final deformed substructure were investigated.

3.7 Microstructure, Dislocation Substructure and Fracture Mechanism Investigation

To analyze the microstructure of specimens after creep test, light metallography was used to determine the changes of grain size, grain boundaries and distribution of coarse carbides MC. The specimens were prepared for light matallography by cutting fractured parts of specimens in longitudinal direction.

Subsequently, cut specimens were mounted in rasin. The mounted specimens were then using silicon carbide papers from 220-1200 grade and polished on cloth wheels using aluminar mixing with water, then using diamond spreays for 0.5 μm, and then immersed in or swabbed with a mixed etchant, namely, Marble etchant, for 5-10 sec, rinsed in water and dried. The Marble etchant had the following chemical composition as follow: 10g.$CuSO_4$, 50 ml.HCl, and 50 ml.H_2O.

To characterize morphology, and nature of gamma prime particles, their size and shape, also the mechanism of deformation and state of grain boundaries, the TEM JEM 2000 FX at accelerating voltage 200 kV was used. The thin foils for TEM were prepared by cutting along longitudinal axis of fractured specimens to make plate-like disc with the thickness of 0.5 mm. Subsequently, the specimecs were ground to the thickness about 0.1 mm. Then thin foils were electropolished at a potential of 25-30 V in solution of $HClO_4$ acid (10% by volume) in methanol. The temperature of solution was controlled to be less than -30°C. After a suitable etching time, the specimens were rapidly removed and washed in methanol and dried. With this electrolytic etching by window technic method, the precipitation particles and dislocation substructure were revealed under crossed polarised light.

Additionally, to examine fracture characteristics, crack nucleation and mode of propagation, the scaning electron microscope (JSM-35 CF) was used at accelerating voltage (25 kV.). The fractured specimens prior to SEM test was cleaned by ultrasonic in acetone.