





Microstructure Analysis of Alloy X-750+ for Spacer Grid Application in BWR Reactor Core

Atom Prob and SEM Analysis of Alloy X-750+ Using Alloy X-750 and 718 as Reference with Different Heat Treatment

Master's thesis in Nuclear Science and Technology

Mas Ipin

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Department of Physics Division of Microstructure Physics CHALMERS UNIVERSITY OF TECHNOLOGY Gothenburg, Sweden 2020 Microstructure Analysis of Alloy X-750+ for Spacer Grid Application in BWR Reactor Core Atom Prob and SEM Analysis of Alloy X-750+ Using Alloy X-750 and 718 as Reference with Different Heat Treatment MAS IPIN

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Cover: Atom Prob Reconstructed image of Alloy X-750+ showing the morphology of secondary γ' particle with Al+Ti concentration treshold of 8.5%.

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Abstract

Due to its durability in hostile environments, nickel-based superalloy X-750 has been used in high temperature (and pressure) applications such as gas turbine, solar thermal power plant, and steam turbine. In nuclear technology, this material is utilized as springs, bolts, and spacer grids in the reactor core. This thesis focuses on the utilization of alloy X-750 as spacer grid in BWR reactor. A previous study showed that a higher iron content of alloy X-750 gives a better corrosion resistance in simulated environment of BWR reactor (5% and 8% wt of Fe content comparison) [7]. Then, a new type of nickel based superalloys was developed by Westinghouse Electric Sweden AB, the so called X-750+ which has dramatically high iron content, i.e. 18% wt. However, corrosion resistant analysis was not performed in this work. Instead, basic microstructure analysis was carried out to give preliminary insight on the physical properties especially in relation to the iron content. Two other samples beside X-750+ were also analyzed as references which are alloy X-750 and 718. All samples were prepared with two type of heat treatments namely the standard heat and two stages heat treatment. The microstructure analysis was divided into two parts, atom probe tomography and Secondary Electron Microscopy (SEM). Samples for atom probe tomography were prepared using electropolishing method to produce needle-shape samples suitable for LEAP 3000 Atom Probe. Meanwhile, SEM analysis was done using JEOL JSM-7800 Prime SEM and LEO Ultra 5.5 SEM. Samples for SEM were polished using grinding paper and liquid diamond complemented by electroetching step to reveal the secondary particles.

Results from atom probe measurement were analysed using proxigram, a 2-D particle distribution calculation provided by IVAS software. This analysis suggests that both X-750 and X-750+ differ slightly in term of particle volume fraction which might indicating that the mechanical properties are also similar. Meanwhile, standard heat treatment tends to give a slightly larger volume fraction for alloy X-750 but the opposite fashion was observed for alloy X-750+. Furthermore, heat treatment difference seemed do not affect the particle distribution across the particle interfaces. 1-D analysis was done for alloy 718 since the tertiary particle (γ'') mostly situated side by side with secondary particle (γ'). This analysis revealed the grain boundaries for all samples. According to SEM analysis, all samples with standard heat treatment revealed more prominent grain boundaries except for alloy 718. However, further investigation should be performed to obtain the chemical compositions across the grain boundaries, such as Transmission Electron Microscopy (TEM) analysis.

Keywords: alloy X-750, microstructure, atom probe tomography, SEM

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1

Introduction

1.1 Background

In order to fight carbon emissions, any means of low carbon technologies have to be pushed forward. Meanwhile, global energy demand is projected to keep increasing in the future [1]. Different fossil-alternative technologies have been developed intensively in different parts of the world and become one of the main topics of political discussions. Nuclear reactors have become the largest contributor to low carbon electricity and for several cases, could be competitive to fossil-based power plants [2]. However, nuclear reactors have been received a big concern, especially after the Fukushima accident. To answer the problems related to safety, generation III/III⁺ type reactors are currently being built throughout the world, which have better fuel efficiency, superior passive safety systems, and low maintenance and capital cost. However, the majority of nuclear power plants currently in operation today are generation II type reactors providing about 11% of world electricity demand [3].

Nickel-based superalloys have been used in several applications with severe environments such as gas turbine, solar thermal power plant, and steam turbine. Different parts of nuclear power plants also utilize this material where high-temperature resistance is critical. One of the components that suffer from high heat flux in nuclear power plants is the spacer grid, a component that holds the fuel rods together inside the reactor core. Figure (1.1) shows a schematic illustration of a typical BWR reactor spacer gird.

Nickel alloy X-750 has been used for more than 30 years in nuclear reactors due to its ability to maintain its mechanical properties at elevated temperature. It is a precipitation-hardened commercial Ni-Cr-Fe alloy designed to have good corrosion properties, resistance to oxidation and high creep rupture strength up to $800^{\circ}C$ [4]. Depending on the heat treatment, alloy X-750 might have different properties in terms of rupture ductility, high-temperature resistance, tensile properties, resistance to stress corrosion cracking (SCC), etc. [4] However, the general corrosion property of this material is not ideal for nuclear reactor environment and contamination of the radioactive isotope Co-58, as a result of Ni-58 activation, can be released into the reactor [5]. Nonetheless, nickel alloy X-750 is still the best material available today for this kind of application.



Figure 1.1: Typical spacer grid (right) inside a fuel assembly (left) of a BWR fuel SVEA-96 Optima. [6]

The characteristic of alloy X-750 that is responsible for its strength at high temperature is its microstructure. The structure of alloy X-750 is face-centered cubic (fcc), also called gamma phase (γ) and the strengthening mechanism is achieved by precipitation of intermetallic $Ni_3(Al, Ti)$, called gamma prime (γ'), phase. Normally γ' - precipitates are embedded in γ -phase, with which they are coherent.

The harsh environment of nuclear reactors is a considerably challenging when it comes to corrosion. It has been reported that a higher iron content provides a better corrosion resistance of alloy X-750 [7]. In that study, an investigation of the oxide evolution of pre-oxidized alloy X-750 was carried out with two different iron contents, 5wt% and 8wt% [7]. The growth of the oxide layer and the metal dissolution were reported to be lower in the higher iron content material

1.2 Scope of this work

The influence of iron content in the previous study gives rise to a prediction that an even higher iron content might lead to even better corrosion resistance of alloy X-750. A new alloy, based on alloy X-750, has been developed by Westinghouse Electric Corporation together with Sandvik Materials Technology, which contains 18% wt of iron, doubled the standard specification of alloy X-750. However, the present study does not focus on the corrosion behavior of the newly developed material in nuclear reactors environment. Instead, this work focuses on how the microstructure differs from the standard X-750. Two type of heat treatments were carried out in this study, namely the standard heat treatment (single stage) which has been used for years in nuclear applications and the two stage heat treatment which has been reported to give worst SCC resistant [8]. To have a better insight into the microstructure,

another nickel-based alloy, the more common alloy A718, was also analyzed. An SEM analysis was also carried out just to give a general idea about the precipitation along the grain boundaries.

1.3 Aim

This study aims to analyze the microstructure of the newly developed alloy X-750+. The comparison indicator would be the size and volume fraction of the particles, concentration of different species across the interface, and qualitative distribution of particles along the grain boundaries. The objectives of this study are to get good understandings on:

- How does the iron content affect the particle size?
- How does the heat treatment affect the particle size?
- How does the iron content affect the distribution of particles at grain boundaries?
- How does the heat treatment affect the distribution of particles at grain boundaries?

1. Introduction

2

Theory

2.1 Precipitation

The first two sections are referred to [9] unless stated otherwise

2.1.1 2.1.1 Phase transformation in solid state

Solid state always assumes the lowest state of Gibbs free energy. Hence, atomic rearrangement might occur to form a crystal structure with the lowest free energy. This atomic rearrangement is called phase transformation, which is classified into two types. If the new phase formed has the same crystal structure but different composition as the parent phase, then this process is classified as decomposition. On the other hand, if the crystal structure and composition of the formed phase are different, it is classified as precipitation. The Gibbs free energy comprise of two terms namely the enthalpy (H) and entropy (S) state of the system as given by equation (2.1)

$$G = H - TS \tag{2.1}$$

where T is given in Kelvin. The enthalpy state is only contributed by the binding energies between the neighboring atoms. When atoms rearrange, there will be an exchange of energy which can be gained, lost, or in special case, no difference. In this special case the binding energy does not depend on the arrangement of the atoms which is called the "ideal solution".

The entropy part reflects the arrangement possibilities of the atoms, and together with the enthalpy term the Gibbs free energy is illustrated by figure (2.1)

Here, the exchange energy is denoted by H_0 . For $H_0 \leq 0$ (left and right picture), the mixture will only have a single minima of the free energy, meaning the system will try to reduce its free energy by forming a single type of phase (homogenous). On the other hand, if $H_0 > 0$ (middle picture), there will be two minima of free energy meaning that any fluctuation when the system is a single phase (c_0) will eventually lead to the formation of two different phases with concentration c_1 and c_2 . Any concentration below c_1 or above c_2 will always produce a single phase since the fluctuations will result in a higher free energy.



Figure 2.1: Gibbs free energy (G_m) illustration plotted with phase mixture concentration (c) for different exchange energy possibilities (H_0)

This illustration applies for decomposition process which is a bit different for a precipitation process. There is no common G_m graph in a precipitation process such as illustrated in Figure (2.1). Rather, each phase has its own Gibbs free energy graph as illustrated in Figure (2.2). However, the same principle also applies to this illustration for the phase transformation, i.e. a two-phase system is only attained for $c_1 < c < c_2$. Furthermore, $c_1 and c_2$ are also referred as terminal solubilities (solvus line) in the phase diagram of the binary system at given temperature T. The solubility of the second phase is usually lower at lower temperature.



Figure 2.2: Graphic illustration of free energy of two-phase system comprised of precipitate (β) and matrix (α)

2.1.2 The kinetics of precipitate formation

If the concentration and temperature required to form a two-phase system are met, the precipitation process is attained by three steps, i.e. nucleation, growth, and coarsening.

The nucleation step is initiated by thermal fluctuations that lead to formation of small embryos of the second phase. In the free energy point of view, there are three components that are responsible for the free energy state of the system, i.e. free energy gain per unit volume during the phase transformation (Δg_u), elastic energy distortion between the precipitate and the parent phase (ϵ_{el}), and the surface energy of the two phases (γ). Equation (2) gives the mathematical formulation of these three components.

$$\Delta G(r) = (-\Delta g_u + \epsilon_{el}) \cdot 4/3\pi r^3 + \gamma \cdot 4\pi r^2 \tag{2.2}$$

The critical radius that needs to be exceeded by the nucleus in order to be stable is given by differentiating Equation (2.2) w.r.t r

$$r_0 = 2\gamma / (\Delta g_u - \epsilon_{el}) \tag{2.3}$$

The elastic distortion energy depends on several components, one of them is the lattice parameter. If both matrix and precipitate have the same lattice parameter, a sphere-shaped precipitate is obtained as the ϵ_{el} term is neglected (i.e. ϵ_{el} is proportional to the lattice parameter difference). However, if the lattice parameter is large, one obtains a plate-shaped precipitate. If the interfacial energy is anisotropic the plate-shape is also preferred.

If the critical radius has been achieved, the precipitates will continue to grow which is controlled by diffusion. For the first short time, the precipitates grow proportionally with $t^{(3/2)}$. However after a long time, when adjacent areas have to compete for the remaining precipitate atoms, the growth decreases exponentially.

Not all precipitates grow and reach equilibrium. In fact, some small precipitates are dissolved while large precipitates continue to grow. This process is called coarsening and is referred to as Ostwald ripening. The driving force of this process is the interfacial boundary energy which is smaller for larger but fewer particles than that of smaller and greater number of particles.

2.1.3 Precipitation in nickel alloys

One can see the morphology of nickel alloys in two parts, the matrix and the precipitates. The crystalline structure of the matrix is austenite; a face-centered cubic solution with a random distribution of different species of atoms termed as gamma phase (γ). The different types of precipitates include:

- gamma prime phase $(Ni_3(Al, Ti))$
- gamma double prime phase $(Ni_3(Nb, V))$

• delta phase $(Ni_3(Nb, V))$

The chemical composition of gamma prime phase (γ') is $Ni_3(Al, Ti)$ acting as the most crucial precipitation hardening of the material. The crystalline structure of γ' phase is in principle the same as the structure of the matrix allowing the coherent relationship at the interface. This coherency inhibits dislocation transformation resulting in excellent mechanical strength. Figure (2.3) illustrates how an aluminum atom in the cube corner is surrounded by three face-centered nickel atoms in this ordered structure. Keep in mind that this aluminum atom can be substituted by a titanium atom. The concentration of Al+Ti combined should be more than 2% for γ' to form. The benefit characteristic of the gamma prime phase includes the possibility of high-volume fraction, up to 50%, high solvus temperature, and yield strength up to 1000 MPa around 900°C [10]



Figure 2.3: Schematic illustration of γ' -phase of nickel alloys [11]

If Niobium or Vanadium are also used as part of the alloying elements, gamma double prime phase (γ'') can form. The chemical formula of this phase is $Ni_3(Al, Nb)$, a body-centered tetragonal crystal structure. This precipitation also contributes to material hardening because it has a semi-coherent relationship with the matrix. However, its usefulness is limited to a temperature of about $700^{\circ}C$ because it starts transitioning into the delta phase at higher temperatures, which then compromises the mechanical strength. This phase is considered metastable and forms in nickel alloys with Nb or V concentration above about 5%wt. Figure (2.4) shows the crystal structure of γ'' -phase. The lattice parameters are approximately a=0.362nm and c=0.742nm.

At elevated temperatures above the γ'' -phase stability, a stable delta phase supersedes the γ'' -phase. Its crystal structure is reported to be an orthorhombic D0 \neg a



Figure 2.4: Crystal structure of Ni_3Nb gamma double prime $(\gamma'')[11]$



Figure 2.5: delta-phase prototype structure[14]

with the same chemical structure as γ'' -phase [10] [12] [13]. The relationship between delta-phase and the matrix is not coherent, leading to a decrease in mechanical strength [10]. The prototype of delta-phase structure is illustrated by Figure (2.5)

2.1.4 Grain Boundaries

Grain boundaries are formed when two grains with different orientations or phases meet. Depending on the misorientation angle between the two grains, grain boundaries are categorized as low and high angle boundaries. Generally, when the misorientation angle is less than 150 the boundaries are categorized as low angle boundaries. They are often comprised of an array of dislocations and their properties and structure depend on the angle of dislocations. In contrary, high angle boundaries are normally independent of the angle and more disordered. In the free energy of grain boundary, the dislocation angles could predict the extent of free energy for low angle boundaries while for high boundaries there is no free energy difference.

One of the important aspects of grain boundaries is related to the mechanical strength of the material. The interfacial area hinders dislocations during plastic

transformation. Thus, an improvement in mechanical strength is usually achieved with a smaller grain boundaries.

2.1.5 Atom Probe Tomography

Atom probe tomography is a powerful tool to analyze chemical compositions of materials. It can give spatial distributions of atoms in near-atom level at almost every region of interest. It has been used for different types of solid material, from metal to ice. The latest atom probe development provides a detection efficiency up to 80% enabling precise detection of small atom clusters in certain regions such as impurities and segregation.

The principle of this method is field evaporation by a high field at a small surface. There are three main components involved in atom probe tomography, as shown in figure (2.3). When a high voltage is applied to the specimen, atoms at the tip surface start ionizing and losing their bonding. The tip radius should be small enough to get enough electric field for ionization. These atoms are then repelled from the tip and accelerated by the electric field, then finally hit the detector. By recording the time of flight (TOF) and the impact position of the ions, the 3D reconstruction can be achieved.



Figure 2.6: Schematic illustration of atom probe tomography [7]

To obtain a high electric field besides increasing the voltage, an ultra-sharp specimen is needed. The formula for the electric field is given by equation (2.1)

$$F = V/(k.r) \tag{2.4}$$

Where F is the electric field (V/m), k is a field factor relating to geometry, and r is the specimen radius (m). Ionization occurs when F is higher than the evaporation field value Fe that depends on the material. Table 2.1 presents the evaporation field values of common metallic materials. Generally, the radius of the tip should not be greater than 100nm, and the voltage ranges between 1kV up to 16kV. As atoms move around as a result of thermal vibrations, the working condition is kept in cryogenic temperature to obtain more precise spatial distributions. Typically, the temperature range between 20-80 K is ideal. Furthermore, because ions can easily interact with atoms during the flight time, and to keep the surface atomically clean, an ultra-high vacuum chamber is needed for up to 10^{-11} torr.

Material $k_{\ell} = 5$	5 Field (V/nm)	Specimen radius (nm)	
		1 kV	14 kV
Al	19	10.5	147.4
Si	33	6.1	84.8
Fe	33	6.1	84.8
Ni	35	5.7	80.0
Mo	41	4.9	68.3
W	52	3.8	53.8
Pt	45	4.4	52.2
Au	53	3.8	52.8

Table 2.1: Typical field evaporation values and working tip radius for different working voltages [15]

Under high electric field conditions, the material suffers from high mechanical stresses sometimes leading to specimen fracture. As the evaporation field Fe is temperature dependent, the value is lower at high temperature, a trade-off between detection accuracy and the specimen fracture risk is inevitable.

A safer way to induce field evaporation of atoms from the specimen is by using short laser pulses. The laser pulse heats the specimen apex for a short time, thereby increasing the temperature and the probability of evaporation. Since the size of the specimen is very large compared to the laser heated area, the laser heat energy is immediately dissipated so the laser only affects locally at the tip apex. This method is often useful to analyze weak materials allowing to operate at lower voltages.

2.1.6 Electropolishing

Electropolishing is one of simplest sample preparation techniques for APT. It is only applicable to conductive materials since the working principle is dissolution assisted by an electric current. The process is started with a suitable form of the material, typically, 0.25x0.25x10 - 15mm square bars, cut from the bulk. To minimize deformation along the cutting area, a low-speed saw is often used. It is suggested to have smaller cross sections to reduce the electropolishing time. It is also necessary to have a circular or square cross section to produce round-shape apex at the end of the electropolishing procedure. One should also remove oxide layers or other contamination such as cutting oil on the surface since they inhibit the polishing process.

Typically, the electropolishing procedure comprises two stages. In the first stage, a relatively strong electrolyte is floated on a dense, inert, immiscible liquid. Hence,



Figure 2.7: Schematic illustration of double layer electropolishing technique

it is called the double layer technique. The specimen acts as the electrode in this setup while the counter electrode is usually made of noble metals such as Au or Pt. The objective of this stage is to etch the middle part to produce a neck-shaped specimen. Its vertical position is adjusted in a way that the electrolyte occupies the region of interest, usually at mid-length of the bar. The applied voltage, electrolyte, and temperature are chosen to remove material rapidly and uniformly from the material. Glycerol is often added to the electrolyte to increase viscosity. The vortex movement of the electrolyte will give a better etching uniformity throughout the surface.

Since the polishing process is more effective in oxygen rich areas, the area closest to the surface, where in contact with air, is etched faster. To avoid this preferential etching, one should gently move the specimen up and down while the etching process takes place. There is no certain thickness one has to achieve in this stage, but a thinner neck specimen reduces the polishing time in the next stage. Figure (2.7) illustrates a typical setup of this first electropolishing stage.

The objective of the latter stage is to produce two sharp needles by breaking the neck shape area with a slow etching process. There is no need to put the specimen in two-layer liquid anymore since the neck is much thinner than the shank. This is done by a simple bath inside a more controlled electrolyte, usually weaker, with an applied voltage. Since there will be a sudden decline in current when the separation happens due to the sudden decrease in surface area, it is possible to electronically monitor polishing current and automatically interrupt the process. Both halves of the rod can be used for APT specimen, assuming the lower part is not damaged.

The electropolishing process is often complemented by pulse polishing, a comple-

mentary stage that is commonly used in an attempt to reveal some feature of interest in the apex [16]. This stage could also remove some residual oxide at the apex, which is often associated with premature specimen failure. Frequently, the same electrolyte is used, and a few short (millisecond) voltage pulses are applied.

2.1.7 Scanning Electron Microscopy

Since the first time this technique was introduced, SEM has been used in many different applications. It offers an excellent image quality in high magnification up to more than 30.000 times [17].

The working principle of this technique is the interaction between an electron beam and matter. Since the atom nucleus occupies only a very small part of the space, electrons will most likely transfer their energy by hitting the electron shell. A heavier atom contains more electron shells resulting in a higher probability that an interaction occurs, which then can be used as identification of material imaging. The key components of SEM consist of the electron beam, scanning coils, and the detector. The electron source could be either a thermionic electron or field emission gun as an electron beam, having typical energy ranges between 1keV to 20keV. The electron energy is chosen based on the x-ray characteristic energy of investigated material. The scanning coils control the scanned area, which then relates to the image magnification. The most common detectors used in SEM are Secondary Electron (SE) detector and Backscatter Electron (BSE) detector. SE detector provides good information about the sample surface since the secondary electrons are produced mainly at the surface while backscattered electrons originate from a larger depth of the material giving elemental contrast. Electron interaction with material is illustrated by Figure (2.8) and the typical schematic of SEM configuration can be seen in figure (2.9).



Figure 2.8: Schematic Illustration of electron interaction with material in different depth (left) and the corresponding energies associated with the type of interaction (right) [18]



Figure 2.9: Schematic diagram of basic components in Scanning Electron Microscopy [18]

Experiments

3.1 Experiment materials, procedure, and methods

3.1.1 Specimen Description

Three type of nickel-based alloys were prepared for this work. The chemical compositions are presented in tabel (3.1). All materials were subjected to two types of heat treatment. The first one is the standard heat treatment used in nuclear applications for many years, which is aging at 700°C for 20 hours while the second heat treatment is considered excellent in other applications. The two- step heat treatment includes aging for 1 hour at 980°C, air cooling to room temperature, then aging at 730°C for 8 hours, oven cooling to room temperature, then aging at 620°C for 8 hours and finally cooled- down to room temperature.

	X750	X750	A718
С	0.03	0.008	0.042
Si	0.35	0.42	0.11
Mn	0.57	0.56	0.04
Р	0.003	0.003	< 0.003
\mathbf{S}	0.002	0.003	0.001
Cr	15.99	15.72	17.99
Ni	70.8	60.7	53.7
Co	< 0.005	< 0.005	0.053
Nb	0.81(+Ta)	0.78	5
Ta	< 0.01	< 0.03	0.02
Ti	2.37	2.39	1
Al	0.59	0.7	0.57
Fe	8.32	18.3	18.39
Cu	< 0.01	< 0.01	< 0.001
Ν	0.01	0.008	0.007
Mo			3.04

Table 3.1: Weight percentage of x-750 (San75), x-750+ (San62), and Inconel 718



Figure 3.1: Sample mounting on brass (a) and a typical setup for slow-speed saw (b)

3.1.2 3.1.2 Sample preparation for electropolishing

All bulk samples were delivered in a square plate specimen with approximately 0.3mm of thickness. The preparation was started by preparing a suitable dimension for the electropolishing step. The samples were cut using a pair of ordinary scissors into 15mm in width prior to low-speed cutting to make rod-shaped specimens, although it is suggested to cut them in a more appropriate way to avoid bending at the edges hence giving an easier mounting. A rectangular brass piece is used to mount the sample by attaching it using ordinary commercial superglue as presented in figure (3.1)a. The sample is then placed on the top part of low-speed saw machine as shown in figure (3.1)b. The blade used for this kind of material was a diamond blade lubricated by iso-cut oil coolant. It is necessary to dress the blade prior to the operation to remove the layer covering the diamond matrix. When the diamond matrix is not exposed enough, the heat of friction can harm the diamond matrix and eventually making the blade dull. Frequently, dressing is performed for every new sample. The sample placement against the blade has to be tangential to make the cutting process more efficient (less cutting on the brass support). To adjust the width of the rod produced, the horizontal position of the sample is moved sequentially by rotating a micrometer screw. The sample was sectioned to 0.3mm in width by turning the micrometer 0.6mm away since another 0.3mm was consumed by the blade. As the cutting surface became larger, more weight was added gradually to make the cutting process faster. After a 0.3x0.3x15mm size rods are obtained, they were then cleaned using Acetone and some thin film of material left during the cutting were removed using scalpel under an optical microscope.

3.1.3 Electropolishing method

Three electropolishing steps were utilized in this experiment. Firstly, neck-shape specimens were acquired using the double layer technique method. The electrolyte layer was made of 10% perchloric acid and 20% glycerol in methanol while Galden



Figure 3.2: A typical setup for two layer electropolishing, a lighter electrolyte on top of Galden

was used as the inert liquid at the bottom. The electrolyte thickness was set just enough to counter the adhesive tension from the beaker while the bottom layer was set to give a comfortable working range for the etching region adjustment. In order to give a better visibility during the electropolishing, a special beaker was used (figure (3.2)), with its top part ground away to remove its curvy part.

The suitable etching voltage of nickel-based alloys was reported to be around 22 V (DC) [15]. To avoid a voltage drop during the electropolishing, the maximum allowed current was set to a reasonably high value. In this experiment, the maximum current was set to 0.54A, which of course in other cases depend on the electrolyte thickness and the rod size.

Unnecessary vibrations of the specimen were not desired due to the specimen fragility. So, instead of moving the sample, the vertical position of the electrolyte was adjusted by seating it on top of a lever. The beaker was then lifted until the electrolyte layer occupied the middle part of the rod (neck-region target area), then the voltage was applied while moving up and down the electrolyte. The process is stopped after a neck-shaped specimen was acquired in which its thickness determined the time needed for the second step.

This time, the specimen was placed in 2% perchloric acid in 2-Butoxyethanol solution. The same voltage was applied using a special power supply that can monitor a sudden decrease in current when the separation took place. The etching time depends on how thin the neck-shape was from the previous step, in this experiment it ranges from about 10-20 minutes. The bottom part of the specimen should be taken out from the electrolyte as soon as possible after the separation to avoid further oxidation on the tip. Both needles were then cleaned using ethanol and prepared for the last step.

The purpose of the last step was to remove remaining oxides on the needle tip by applying short voltage pulses in the same electrolyte solution. In this experiment, 10 pulses were given at 5 ms pulse width and 22-volt amplitude.

3.1.4 Sample Preparation for SEM

Initially, all samples were cut into approximately 0.03x1x1.5 cm as a suitable size for mounting for SEM. The sample was then clamped vertically with a stainless steel holder. After that, they were buried together under melted conductive epoxy, until finally a solid cylinder was obtained after it cooled down. The next step was to polish the cylinder cross section with multiple stages to reveal the material microstructure. The polishing step includes SiC-paper grinding, liquid diamond polishing, and colloidal silica. For the paper grinding purpose, papers of 220, 420, 600, and 1000 grits were used subsequently for 2-4 minutes grinding time each. Similarly, liquid diamond of 9, 3, and 1 micron were used for the next polishing step. The polishing stage was ended with colloidal silica to give some chemical etching.

After a smooth surface is achieved by the mechanical etching, the sample is then etched electrolytically (called electroetching process) in order to reveal the grain boundaries precipitates at the grain boundaries, and hopefully the gamma prime particles. The technical setup is explained as follows. To produce the electrolyte, four grams of oxalic acid was diluted in 50 ml of water. Four volt of DC voltage was then applied for about 5 seconds to perform the etching process. This step was performed to reveal the γ' particles in Hynes 282 superalloys in a previous study [19].

3.2 Data Acquisition

3.2.1 Atom Probe Analysis

A LEAP 3000X HR (Imago Scientific Instruments) was utilized in this experiment with the following working conditions. The chamber pressure was stabilized as low as possible, something between 10^{-10} to 10^{-11} torr, containing primarily hydrogen as residual gas. The cryogenic temperature was set differently between the voltage pulse mode and the laser pulse mode, 70K and 50K, respectively. The instrument is equipped with two temperature sensors; one located close to the specimen while the other is situated closer to the cryo-head. Hence, there is always a margin reading between the two, typically around 10K, and the ideal condition is considered when they are stable.

All samples were run in voltage mode except for one, i.e. the two stage-heat treatment alloy 718. There was no particular reason for this analysis mode other than in attempt to get enough data since all specimen for this material fractured early. The pulse was set to 200kHz in both modes with the same evaporation target, i.e. 0.2%. The LEAP 3000X HR is equipped with a green laser ($\lambda = 535$ nm), and the energy was set to 0.3nJ. It is also equipped with an energy compensating reflectron, improving the mass resolution in voltage mode.

The detector collision history was recorded in so-called RHIT file which was then transferred to IVAS software for evaluation. To visualize the chemical distribution of the consumed apex during the measurement, one creates concentration profile of a certain species in form of iso-surfaces. The term iso-surface describes points in which the concentrations of intended species are equal. The species was chosen to represent phases in metallic structure, i.e. Al + Ti and Nb representing the γ' -phase and γ'' -phase, respectively.

To quantify the chemical distribution across the interface, one uses proxigram which is a built-in calculation tool of IVAS. With the help of iso-surface obtained previously, the species concentration is calculated across the surface inwards and outwards within the specified range. The calculation is performed for all the surfaces and the average values are taken. Proxigram works fine on double phase material where the surfaces are surrounded homogenously. Otherwise, 1-D concentration profile is preferred because one can control the region of interest.

3.2.2 Scanning Electron Microscopy

JEOL, JSM-7800 Prime SEM and LEO Ultra 5.5 were used in this experiment in secondary electron mode and backscattered mode. The electron beam energy was set to 15keV with 106 uA of current. For nickel-based alloy, 15keV was high enough to cover all the x-ray characteristics of the elemental species in the material.

To acquire good images from SEM, several aspects should be considered beside the electron energy and current. The first parameter is the working distance. The working distance in which the electron beam is focused depends on the SEM machine and the type of detector used. For JEOL, JSM-7800 the working distance is found to be typically 9.1 mm and 3.9 mm for secondary electron detector and backscattering detector respectively. However, these values might vary based on the accuracy of the thickness measurement of the sample prior the acquisition.

There are two type of detector used in LEO Ultra 5.5 as well which based on the secondary and backscatter electron interaction. However, unlike the JEOL, this machine uses what so called In-lens detector for the secondary electron detector in which is believed to have a better sensitivity on the surface feature [20]. The working distance for this machine was found to be between 8-10mm and 12mm for In-lens detector and backscatter electron respectively.

The second parameter in the SEM acquisition that also important to consider is the astigmatism parameter. The effect of incorrect setting for the astigmatism is blurry and elongated images due to the non-uniform magnetic fields. The cause of astigmatism is due to the sample misalignment, lens imperfectness, contaminations, and non-uniform magnetic field of the lens. The stigmator correction is done by compensating the non-uniform magnetic fields in the lens. The value of course depends on the object placement and the detector type so that different configuration is required for each samples and detector type used.

The last parameter to consider is the Wobble parameter. This parameter is adjusted via x and y axis to make sure that the beam and the lens is perfectly perpendicular.

4

Results and Discussion

4.1 Effect of Different Heat Treatments on Particle Volume Fraction

This section will be started by recalling the two types of heat treatments performed in this experiment, one was the standard heat-treatment used in nuclear industry $(705^{\circ}C \text{ for } 20 \text{ hours})$, and the other one was the two-stage heat treatment as described in section 3.1.1.

The particle morphology can be illustrated by drawing an iso-surface of elements that compose the particle of interest. In this case, the γ' -phase can be illustrated by the iso-surface concentration of Al or Ti (or the sum of the two), while the γ'' -phase can be shown by Nb. One cannot achieve very clear contrast of the interface between the particle and the matrix due to some aberrations in the APT measurements. This is caused by so called local magnification effects (resulting from the two phases having different evaporation fields) and there is also a risk that ions move on the surface before the evaporation, which is more common when using laser pulse mode (and affecting some elements like for example Si). Hence, a gradual, diffuse profile is inevitable. There is no absolute correct threshold one can chose to represent precipitates accurately due to this bias, making the size determination challenging.

Visually, as shown in Figure (4.1) the particle size between the two heat treatments is not significantly different. Please keep in mind that a higher threshold gives smaller particles in the images, as the measured concentrations of Al and Ti are higher towards the center. Thus, it is important to set the threshold at the same value for all material conditions for comparison. Furthermore, since the evaporation field of the particles is higher than the matrix, the particles appear bigger than the actual size due to local magnification [21].

One way to calculate the volume fraction of the particles is by comparing the number of ions obtained by the APT and taking into account the lattice parameter of the matrix and particles, respectively. One could argue that the accuracy of this calculation relies substantially on the number of ions collected by the APT due to the size distribution of particles. To tackle this problem, several measurements treated in the same manner could reduce this statistical inaccuracy. However, having more



Figure 4.1: γ' -phase morphology of alloy x-750 for standard heat treatment (a) and two-stage heat treatment (b) with iso-surface based on the threshold of Al + Ti concentration at 8.5% at

specimen measured is not always practical especially when the specimens fracture easily. Furthermore, data with more ions are always more reliable than those with fewer one.

Figure (4.2) shows that the particle volume fraction of alloy X-750 is slightly lower for the two-stage heat treatment. At this point, one cannot confidently draw any conclusion regarding the mechanical properties but could say that they might be very close considering these small differences.

Regarding the other two materials, i.e. alloy X-750+, the same particle size analysis was done and presented in figures (4.3-4.4). Alloy X-750+ exhibits a different fashion regarding the particle volume fraction for the standard and two step heat treatment. In contrast to X-750, two stage heat treatment gave a slightly higher particle volume fraction than the standard one. However, the difference is still too small to give a prediction regarding the mechanical properties.

The morphology of precipitation in alloy 718 is shown in Figure (4.5). The figure suggests that γ' and γ'' coexist in the same particles, split in half. The particle volume fraction can be obtained by drawing iso-surfaces of Al+Ti+Nb together, treating both phases as one particle. Once again, due to the surface bias, the volume fraction was analyzed for three different threshold values as shown in Figure (4.6).

4.2 Element Concentration Across the Interface

This section focuses on how different species are distributed across the interface between the matrix and the particle. Besides the obvious aspect affecting this distribution, i.e. the chemical composition specified for each material, it is also interesting to see how the heat treatment plays a role. For the dual phase system (γ/γ') , the



Figure 4.2: Particle volume fraction of alloy x-750 for standard heat treatment (SHT) and two stage heat treatment (BHT)



Figure 4.3: γ' -phase morphology of alloy X-750+ for standard heat treatment (a) and two stage heat treatment (b) with iso-surfaces based on the threshold of Al + Ti concentration at 8.5% at.



Figure 4.4: Particle volume fraction of alloy X-750+ for standard heat treatment (SHT) and two step heat treatment (BHT) with different Al+Ti threshold values.



Figure 4.5: The morphology of γ' -phase (blue) and γ'' -phase (brown) in alloy 718 for standard heat treatment (a) and two stage heat treatment (b).



Figure 4.6: Particle volume fraction in alloy 718 for standard heat treatment (SHT) and two stage heat treatment (BHT).

interface species concentration can be presented by proxigram graphs provided by the IVAS program. On the other hand, a proxigram is meaningless for the overlapping phases that might appear in triple phase systems $(\gamma/\gamma'/\gamma'')$ like alloy 718. For convenience, all graphs are grouped as major elements and minor elements groups to give a better clarity.

Based on Figure (4.7), it appears as if there is no significant difference in terms of species distribution regarding the two different heat treatments. Most of Al+Ti are located inside the particles as expected, together with an increase in Ni due to the depletion of Fe and Cr. Si and Mn segregate to the interface in which the two-step heat treatment gives a higher magnitude, in particular for Si. Meanwhile, C prefers to be inside the particles and also Nb is slightly enriched in the particles.

A similar fashion is also observed in alloy X-750+ as shown in Figure (4.8). We can immediately see a high Fe concentration in the matrix of alloy X-750+ as expected. Since most of Fe concentrate in the matrix in X-750+, it is a good indication that the Fe addition does not affect much in terms of mechanical properties. The difference of heat treatment exhibits no influence on the species concentration profiles Based on Figure (4.5), it is hard to determine the species concentration across the interface of alloy 718 using proxigram analysis. Hence, a 1-D concentration profile is drawn across the phase interfaces in one particular particle employing the Region of Interest (ROI) feature provided by the IVAS program. The ROI schematic and the concentration profiles are presented side by side in Figure (4.9) and 4.10).

According to Figure (4.9) and 4.10), there is no significant difference in terms of the distribution of the species across the particle interface. The size of gamma double prime in the standard heat treatment appears bigger, which is not always the case but depends on the ROI selection. A similar trend is observed for the Ni



Figure 4.7: Atomic concentration across the interface of major (top) and minor (bottom) elements of X-750 for both standard (SHT) and two stage heat treatment (BHT) at 8.5% Al+Ti threshold. Positive offset values represent the inside of the particle while negative values represent the outside. Continuous and dashed lines indicate the type of heat treatment



Figure 4.8: Atomic concentration across the interface of major (top) and minor (bottom) elements of x-750+ for both standard (SHT) and two-stage heat treatment (BHT) at 8.5% Al+Ti threshold. Positive offset values directing inwards the particle while negative offset directing outward.



Figure 4.9: Atomic concentration across the interface of alloy 718 for standard heat treatment.



Figure 4.10: Atomic concentration across the interface of alloy 718 for two-step heat treatment.

concentration that is higher in the particle than in the matrix due to Cr and Fe depletion. Furthermore, the gamma double prime phase contains a slightly higher nickel concentration. It is also observed that there is an overlapping region between the two particles where the thickness cannot be determined due to the interface bias. Lastly, Nb is also present in the gamma prime particle at a similar concentration as Al+Ti appear in gamma double prime phase, both about 5% at.

4.3 Scanning Electron Microscope Analysis

4.3.1 4.3.1. JEOL, JSM-7800 Prime SEM

This section presents SEM images for the two heat treatments side by side. In this part, the magnification was not high enough to reveal the γ , γ' , or γ'' particles clearly but gives sufficient idea of the features across the grain boundaries.

According to Figure (4.11), some big particles appear only in the standard heat treatment for alloy X-750, with the same fashion also observed in alloy x-750+ as shown in Figure (4.12). On the other hand, a standard heat treatment of alloy 718



Figure 4.11: SEM image of grain boundary of alloy x-750 with standard heat treatment (a) and two-stage heat treatment (b).



Figure 4.12: SEM image of grain boundary of alloy x-750+ with standard heat treatment (a) and two-stage heat treatment (b).

produces smooth grain boundaries without any particles while plate shaped niobium rich delta phase [22] particles are formed in the grain boundaries with the two-stage heat treatment (Figure (4.13)).

4.3.2 LEO Ultra 55

In order to get a better idea of the features across the boundaries a further analysis was done using LEO Ultra 55. The same samples were prepared the same way except that one additional step was done, which was an electroetching step. The following figures present the results using the LEO Ultra 5.5 with the electroetching step.

For superalloy X-750 and X-750+ the same fashion is observed for both standard and



Figure 4.13: SEM image of grain boundary of alloy 718 with standard heat treatment (a) and two-stage heat treatment (b).



Figure 4.14: SEM image of grain boundary of alloy x-750 with standard heat treatment (a) and two-stage heat treatment (b).



Figure 4.15: SEM image of grain boundary of alloy x-750+ with standard heat treatment (a) and two-stage heat treatment (b).



Figure 4.16: SEM image of grain boundary of alloy 718 with standard heat treatment (a) and two-stage heat treatment (b).

two-stages heat treatment. We can see that the standard heat treatment produces grain boundaries that are more visible. It seems that the standard heat treated samples were easier to etch than the two-stage heat treated ones. The similar results were also observed in the previous section, i.e. the grain boundaries of the standard heat treated materials were also more visible, see Figure (4.14) and (4.15). One could say that the grain boundaries of the standard heat treated materials are thicker than those of the two-stage heat treated ones. Please note that the thickness of grain boundaries of the two-stage heat treated samples appears wider due to the etching. Meanwhile, superalloy 718 exhibits the opposite fashion with the two-stage heat treated sample showing more contrast of grain boundaries, see Figure (4.16). This observation also agrees with the SEM results from the previous section, i.e. Figure (4.11) to (4.13).

Generally, the features across the grain boundaries are more visible compared to the ones without the electroetching step. However, it is still hard to identify the type of particles across the grain boundaries due to the samples were probably overetched. A further investigations using TEM might be useful to identify the chemical compositions across the grain boundaries for a better understanding about the effect of the heat treatment and the iron content. Lastly, employing the electroetching steps did reveal the secondary particle as shown in Figure (4.17) below.



Figure 4.17: Secondary particles in electroetched sample alloy X-750+ using LEO Ultra 55 SEM instrument.

Conclusion

Atom probe tomography analysis was performed to obtain the microstructure characteristics for different materials and heat treatments, especially for alloy X-750 and X-750+. The iron addition in alloy X-750+ exhibits only a slight difference in terms of particle morphology compared to alloy X-750. Furthermore, in terms of volume fraction, the standard heat treatment gives a lower precipitate volume fraction in alloy X-750 while the opposite trend was observed for alloy X-750+. However, no noticeable differences were observed in terms of element distribution across the particle interfaces.

The same analysis was also performed for alloy 718 as a comparison. Secondary and tertiary particles were observed for both heat treatments. which mostly stick to each other. The presence of tertiary particles (γ'') in alloy 718 makes it difficult to perform proxigram analysis. The particle distribution calculation was done using 1-D analysis suggesting no significant differences between the standard and two-stage heat treatment.

SEM analysis was performed using two SEM instruments. The results suggest that grain boundaries were more visible for the standard heat treatment in alloy X-750 and X750+ while the opposite fashion was observed in alloy 718. A further investigation with TEM analysis might be useful to obtain chemical composition across the grain boundaries for a better insight on the effect of iron content addition and the heat treatment difference. Lastly, the electroetching step was successful in revealing the secondary particle in alloy X-750+.

5. Conclusion

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