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EFFECT OF RE-HEAT TREATMENT CONDITIONS ON MICROSTRUCTURAL REFURBISHMENT OF NICKEL BASED SUPERALLOY TURBINE BLADES, IN-738, AFTER LONG-TERM SERVICE

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ABSTRACT

The effects of re-heat-treatment conditions on the microstructure of cast polycrystalline nickel based superalloy, IN-738, operated by the Electricity Generating Authority of Thailand (EGAT) for long-term service (70,000 hrs) were investigated. It was found that the microstructure of the exposed specimen could be recovered by a re-solution treatment followed by a two-step aging treatment. During solution treatment, the coarse carbides and gamma prime precipitates were dissolved into the matrix. Then specimens were treated through a series of aging resulting in a uniformly dispersed precipitation of gamma prime particles, which is more uniform than those in the long-term exposed microstructure. However, it was also found that the higher solution annealing temperature resulted in less volume fraction of gamma prime precipitates and a less homogeneous microstructure than the lower one.

Keywords: Microstructural Refurbishment, Rejuvenation, Re-Heat-Treatment, Superalloys, Lifetime Extension, IN-738

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INTRODUCTION

This paper presents a route of applied research and development, which is possible to be done in Thailand for the rejuvenation of superalloy components in a land-based gas turbine industry especially power plants in Thailand. The increasing of usage and success of the gas turbine power-generation industry in Thailand is strongly dependent upon satisfying the demands of a range of customers and operators and by arising from a number of market and legislative factors such as capital costs, operational costs, efficiency, power output, fuels, emission (to environment) and so on. Therefore, the operators or electricity generating producers with their own maintenance and repairing, need a wide range of materials engineering knowledge and skills to ensure safety, reliable operation of the engines as well as costs. However, in Thailand, there are still very few applied research and developments related to this field. The country, by electricity producers, has lost lots of money every year to purchase new expensive superalloy components from abroad and/or has to dispatch long-term serviced components to abroad for repairs. Therefore, the present work expresses the simple and possible routes for repairing superalloy components in Thailand.

Gas Turbine Engines

There are three main sections in gas turbine engines: compressor, burner and turbine; see details of the engine in Figure 1. The gas turbine engine compresses the ingested air from the atmosphere several times in the compressor section. Adding fuel and burning mixture in the combustion section results in turbine inlet gases in the temperature range of 730 to 1730°C. The outcome is high-pressure hot gas steam, which rotates a turbine, which in turn drives the compressor. The remaining hot gas is useful for thrust producing in turbine engines or shaft horsepower in turbo shaft engines. As it is very clear to understand that the components in the gas turbine engines must be made from special metallic materials, which can withstand long-term service at elevated temperatures. Such materials require not only high strength but also, adequate ductility to tolerate creep deformation during operation, to resist low-cycle fatigue deformation (especially dynamic components) as well as good hot oxidation and hot corrosion resistances. However, coating can be often applied to improve these surface degradation resistances. Superalloys, which consist of many elements inside, were found to fulfill all the requirements above.

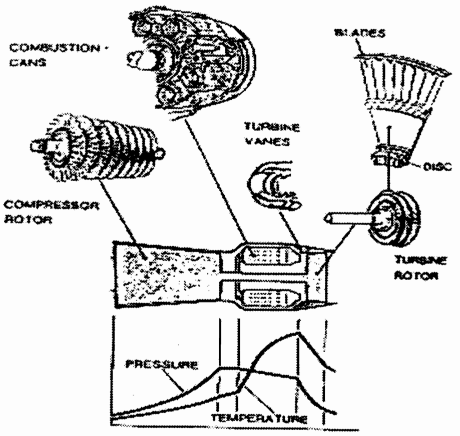

Figure 1 Hot section of a gas turbine engine with relative temperature and pressure profile. (Sims and Hagel, 1972)

Superalloys

Superalloys are generally used as gas turbine components operating at temperatures above 550°C and up to about 1200°C, which include ducts, cases, and liners as well as for major components such as turbine blades, vanes, disks, and combustion cans. Superalloys can be classified into three main groups: 1) Cobalt-based, 2) Nickel-based and 3) Iron-based superalloys. However, Nickel-Iron-based superalloys are considered a special group within the nickel-based group. Chromium and Titanium alloys are not regarded as superalloys. Superalloys are now used in aircraft, marine, industrial and vehicular gas turbines, space vehicles, rocket engines, experimental aircraft, nuclear reactors, submarines, steam power plants, petrochemical equipments, and other high-temperature applications. The largest use of superalloys in the present day is in the gas turbine industry for aircrafts (Figure 2) and power plants (Sims, *et al.* 1972; Tien, *et al.* 1989; Sims, *et al.* 1987; Bradley, 1988; and Davis, 2000). In contemporary engines, nickel-based alloys are used as dynamic turbine blades, nickel- or nickel-iron-based alloys as turbine wheels and nickel or cobalt-based alloys as vane and combustion cans.

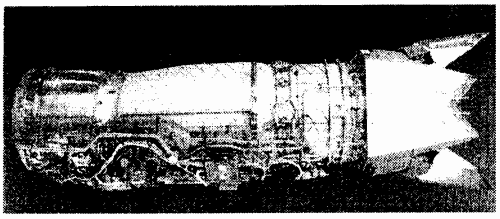

Figure 2 Gas turbine engine-a major user of superalloys. (Donachie and Donachie, 2002)

Superalloys, which can be used to withstand high stress (load) at elevated temperatures, generally have high creep resistance, high temperature fatigue resistance (both low and high cycles), good thermal fatigue resistance (low thermal expansion and high thermal conductivity) as well as high strength and ductility. Their microstructures and mechanical properties should be stable during operating at high temperatures for long-term service. Furthermore, they should resist surface degradation by hot oxidation and hot corrosion. The other minor requirements are good weldability and formability (in the case of wrought polycrystalline alloys).

Superalloys are the materials, which have outstanding strength over high temperatures. The Face-Centered-Cubic (FCC) lattice of austenite composition has a great capability to maintain good tensile, rupture and creep properties at high temperatures, which is much better than equivalent Body-Centered-Cubic (BCC) systems (Sims and Hagel, 1972). A very important feature is its solubility of many other elements in the austenitic matrix and ability to control the precipitation of intermetallic phases such as gamma prime and/or double prime (γ' and/or γ'') for excellent strength, in the case of nickel- and cobalt-based-alloys. Strength in superalloys can be principally obtained by:

1) Precipitation strengthening by intermetallic phases (γ' in Ni-base alloys and γ'' in Ni-Fe-base alloys).

2) Solid solution strengthening.

3) Carbide precipitation (both in matrix and on grain boundaries), especially in both wrought and cast polycrystalline alloys but not in single crystal superalloys.

In the present work, nickel-based alloys are emphasized and concerned with because they are the material components, which are mostly used and retired in gas turbine industries in Thailand. Therefore, more information about nickel- and/or nickel-iron-based alloys will be discussed in the paper. The microstructure of nickel-based alloy is presented in Figure 3. Nickel based alloys consist of many elements included nickel, chromium, cobalt, molybdenum, tungsten, tantalum, columbium, aluminum, titanium, iron, manganese, silicon, carbon, boron,

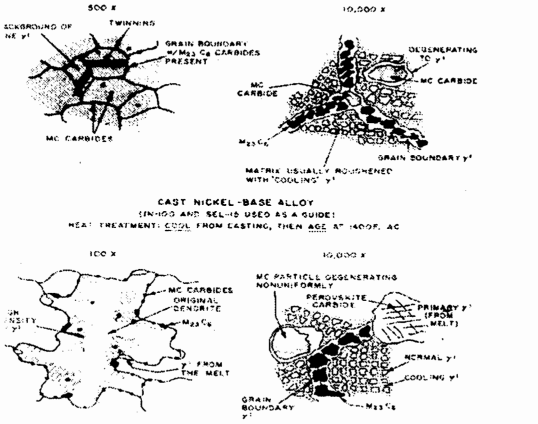

Figure 3 Microstructure of nickel-based alloy (Sims and Hagel, 1972)

zirconium, and other less important elements. From a periodic table, the elements can be divided in three main groups:

1) Elements, which form FCC matrix, come from group 5, 6, and 7 that are nickel, cobalt, iron, chromium, molybdenum, tungsten, and vanadium.

2) Elements, which form gamma prime precipitation of Ni_3Al, come from group 3, 4, and 5 that are aluminum, titanium, niobium, and tantalum (titanium, niobium, and tantalum can substitute aluminum in Ni_3Al.

3) Elements, which introduce segregation on grain boundaries, are magnesium, boron, carbon, and zirconium.

Table 1 reviews briefly the role of various alloying elements play in strengthening conventional nickel-based-alloys.

Table 1 Role of alloying elements on nickel-based-alloys.

Effects	Elements
- Solid solution strengtheners	Co, Cr, Fe, Mo, W, Ta
- Carbide form:	
- MC type	W, Ta, Ti, Mo, Nb
- M_7C_3type	Cr
- $M_{23}C_6$type	Cr, Mo, W
- M_6C type	Mo, W
- Carbonitrides: M (CN) type	C, N
- Oxidation resistance	Al, Cr
- Hot corrosion resistance improvement	La, Th
- Sulfidation resistance	Cr
- Improves creep properties	B
- Increase rupture strength	B
- Causes grain boundary segregation	B, C, Cr

The individual phases in the microstructure of superalloys have a tendency to transform toward equilibrium when exposed to high temperatures. Many phase changes could occur depending on the temperature levels and time of exposure. Unfortunately, phase instability results in weakened- or brittle- phase formation presence in some alloys. Nickel is an excellent base metal when it is highly alloyed. It has a stronger tolerance for alloying additions without any detrimental phase formation. Furthermore, after long-term exposed services without or with applied load (especially under creep conditions, which is regarded as the most widely operated condition), precipitates could become coarsened and coalesced, which could be followed by void formation on grain boundaries (Sims, *et al.* (1972), Tien, *et al.* (1989), Sims, *et al.* (1987), Bradley, (1988), Davis, (2000), Donachie, *et al.* (2002), Donachie, (1984), and Meetham, *et al.* (2000), as seen in Figure 4.

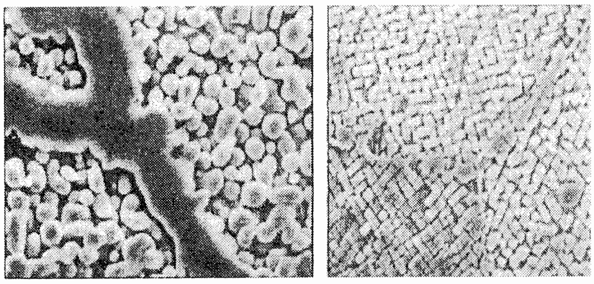

Figure 4 Microstructure after long-term service and rejuvenated microstructure of GTD111. (www.liburdi.com)

Such kind of degraded microstructure would then theoretically provide worse mechanical properties at elevated temperatures resulting in a decrease of lifetime services. Not only phase changes occurring under applied load at high temperature conditions, microvoids or creep cavitations as well as microcracks could appear intergranularly and/or transgranularly in the alloys causing the following nucleation and propagation of macroscopic fracture. Therefore, the main concept for material repair routes is to eliminate the creep damage by closing microvoids or microcracks including restoring the alloy microstructure by hot isostatic pressing (HIP) and rejuvenation heat-treatment processes to reach the new material properties (creep strength) (Tien, *et al.* 1989; Sims, *et al.* 1987; Bradley, 1988; Davis, 2000; Donachie, *et al.* 2002; Donachie, 1984;, and Meetham, *et al.* 2000).

Moreover, mechanical damage of the superalloy blades or vanes could occur from foreign object damage, rubs, erosion, and burning. Depending on the location and severity of the damage, the repairs can be completed by a process such as blending, precision welding, plasma spray, or powder metallurgy (PM). Any type of this macroscopic damage must be repaired before the alloy microstructure and mechanical properties of the alloy component being restored. In this paper, the possible routes for applied research about microstructural rejuvenation, which are carried out in Thailand as a collaborative among Metallurgy and Materials Science Research Institute (MMRI), Chulalongkorn University, National Metals and Materials Technology Center of Thailand (MTEC), and Electricity Generating Authority of Thailand (EGAT) for the rejuvenation of reheat-treatment processes, are mentioned below.

Due to its high production cost from complex process conditions and expensive alloying elements being used, the service life extension of gas turbine components is becoming increasingly important in the current days. Therefore, it is very important to establish the methodology of rejuvenation for cast superalloy components after long-term services at high temperatures causing microstructural degradation as well as creep damage. In the case of conventionally polycrystalline cast superalloys, the creep damage occurs firstly as the microstructure changes such as coarsening and coalescence of γ' and/or other precipitates, which is followed by void formation at grain boundaries.

Rejuvenation heat-treatment processes.

Vacuum heat-treatment is widely used during the repair process for many reasons such as improving weldability, stress relieving in welds, coating diffusion and restoring alloy microstructure. In a vacuum furnace, the alloy blades or vanes are held at the solution temperature, which causes the coarse carbides, gamma prime and gamma double prime, being formed in the grain boundaries during service, to be dissolved into the matrix, see Figure 5. The turbine blades or vanes are then processed through a series of aging cycles, which re-precipitate the strengthening phase to form microstructure similar to a new part. Although the perfect microstructure recovery is not always possible due to the presence of grain boundary strengthening elements, i.e., carbon and boron, which decrease the incipient melting temperatures to prevent perfect solutioning of the gamma prime phase.

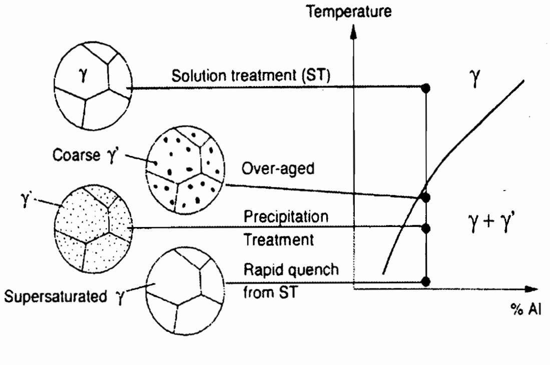

Figure 5 Heat-treatment diagram for precipitation strengthening in nickel-based superalloys. Meetham, and Van de Voorde (2000)

Conditions of heat-treatment processes were developed specially for rejuvenation of each alloy. However, very few applied research programs (Sims, *et al.* (1987), and Bradley, (1988) for rejuvenation heat-treatment has been developed and carried out in Thailand, which have the aim to reduce material costs in the gas turbine industry. During the repairing process for components, sample materials should be subjected to the same heat-treatment and subsequently receive qualification tests such as microstructural examination as well as mechanical tests (hardness, tensile tests at elevated temperatures including creep and/or stress rupture tests to ensure rejuvenated materials meeting the original new material specification.

IN-738, a cast nickel based superalloy was originally developed as a material for turbine blade applications. It was specially designed for long-term service in excess of 100,000 hours at temperatures up to 980°C with surface coating. Its chemical composition was developed to obtain an excellent combination between mechanical and physical properties such as high temperature tensile and fatigue strength, creep strength, fracture toughness, thermal fatigue, structural stability including hot corrosion and hot oxidation resistance. Optimum creep properties, a major concerned application, were achieved by solid solution strengthening in matrix, precipitation strengthening by γ' phase and partially obtaining from carbide phase strengthening, which all are followed by the heat treatment process (solutioning treatment and precipitate aging). The IN-738 material is used as a material for gas turbines in power plants of the Electricity Generating Authority of Thailand (EGAT) in the present day.

It is very necessary to determine methods providing proper microstructural characteristic restoration of the material after long-term usage. The method should also prevent or prolong the service time before creep damage occurs. However, the complete microstructure recovery is not always possible due to the presence of grain boundary strengthening element, i.e. carbon, which decreases the incipient melting temperatures preventing the complete solution of carbide phase. Thus, microstructural degradation before creep damage in cast polycrystalline-nickel base superalloy, IN-738, can be expected to be almost eliminated by a simple re-heat-treatment to restore the proper microstructure for good creep resistance resulting in a longer lifetime service of the material. In the present study, the effect of re-heat-treatment conditions on the microstructural characteristics of nickel based superalloy was examined for the microstructure recovery and the creep life extension.

Experimental Materials and Procedures.

The cast polycrystalline nickel-based superalloy IN-738 was selected as an experimental material. This superalloy is suitable for the manufacturing of turbine blades of gas turbine engines in the power plant, operated at temperatures of up to 980°C with surface coating. The chemical composition of the alloy in weight % is shown in Table 2. The programs of heat treatment conditions, which were carried out for the alloy after a 70,000-hour service are shown in Table 3. The purpose of the present study is to determine the most suitable and practicable heat-treatment condition including solution annealing and precipitate aging, which can provide the best microstructural characteristics. Finally, micro hardness tests were performed randomly to determine the effect of re-heat treatment conditions on hardness.

Table 2 Chemical composition in weight % of IN-738.

Ni	Cr	Co	Mo+W	Al+Ti+Ta+Cb
61	16	8.5	4.4	9.5

Table 3 Heat treatment conditions applied to long term exposed IN-738.

No.	Solution Treatment	Primary precipitate aging	Secondary precipitate aging
1*	1125°C/ 2hr. (AC)	-	845°C/ 24 hr. (AC)
2	1125°C/ 2hr. (AC)	925°C/ 1hr. (AC)	845°C/24 hr. (AC)
3	1125°C/ 2hr. (AC)	1055°C/ 1hr. (AC)	845°C/24 hr. (AC)
4	1175°C/ 2hr. (AC)	-	845°C/24 hr. (AC)
5	1175°C/ 2hr. (AC)	925°C/ 1hr. (AC)	845°C/24 hr. (AC)
6	1175°C/ 2hr. (AC)	1055°C/ 1hr. (AC)	845°C/24 hr. (AC)

* *Standard Heat-Treatment condition*

RESULTS AND DISCUSSION

Microstructure

It was found that the microstructure of the exposed specimen after long-term service (70,000 hrs.) is heterogeneous concerning high concentration of γ' precipitates in many areas. These γ' precipitates are coarsening and coalescing, and have bigger sizes than those in the lower density zone, Figure 6. This type of microstructure is theoretically expected to have a low efficiency to block dislocation movements during loading at high temperatures resulting in lower creep resistance. Therefore, it is needed to recover microstructure to the same as or similar to the original one by a simple re-heat treatment process.

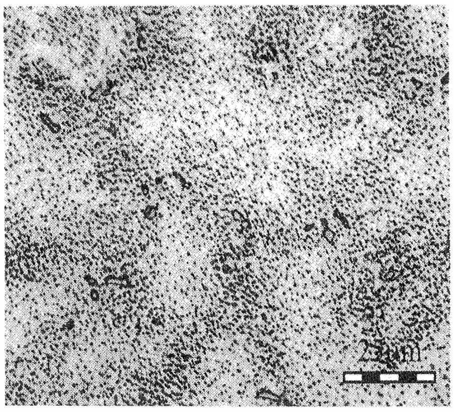

Figure 6 As-received microstructure after long-term service.

According to the previous work www.liburdi.com, repeating the standard heat treatment sequence does not always work well. It is reported that the microstructure was only partially recovered by such simple re-heat treatment. However, when re-heat treatment conditions, according to No.1 (standard heat treatment), were applied to long-term exposed specimens in this study, a much more homogeneous microstructure consisting of a uniform dispersion of gamma prime precipitates are found. The γ' precipitates are very fine and are a similar size and shape, Figure 7. This type of microstructure is theoretically expected and desired as the best microstructure, which can provide very good mechanical properties at elevated temperatures especially both short-term strength at elevated temperatures as well as high creep resistance. However, gamma prime precipitates with very fine and medium sizes can be seen as well.

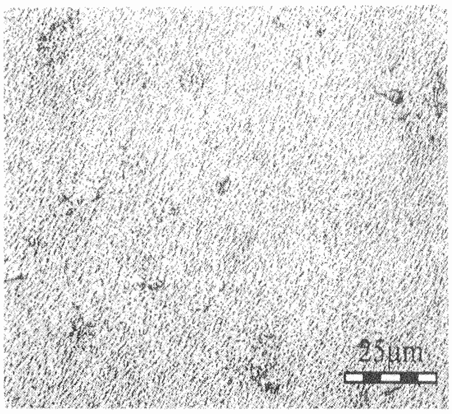

Figure 7 After standard heat-treatment at 1125°C / 2hr. (AC) and 845°C / 24 hr. (AC).

According to heat treatment of program No.2 (Figure 8), primary precipitate aging at 925°C for 1 hour resulted in an early precipitation of gamma prime γ', which its volume fraction is less than that of the microstructure according to program No.1. After secondary precipitate aging, elements needed to form γ' precipitate would diffuse into the former gamma prime precipitates causing the coarsening of the precipitates during secondary aging. It should be noted that the amount of remaining precipitate forming elements might be not enough and/or very low or no driving force to reform very fine gamma prime precipitate during secondary precipitate aging resulting in such kinds of microstructure. Thus, large irregular γ' particles are created at 845°C. Such kinds of microstructure could probably provide good rupture and/or creep resistance. As it is already well known that creep strength of alloys by γ' precipitation is a function of γ' particle size. It can be concluded that the temperature of 925°C as primary aging produced more uniform and coarser rounded γ'.

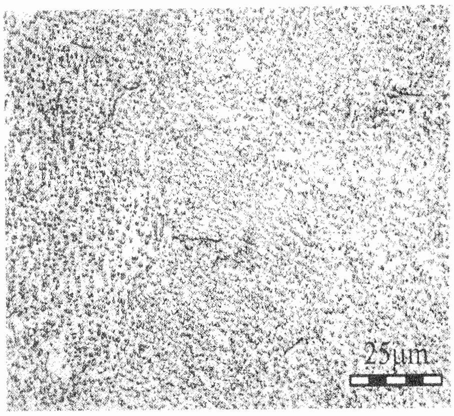

Figure 8 After heat-treatment at 1125°C/ 2hr. (AC), 925°C / 1 hr. (AC), and 845°C / 24 hr. (AC).

In Figure 9, the microstructures after heat treatment according to program No.3 show the uniform dispersion of gamma prime precipitate. The microstructure is very similar to the microstructure of sample No. 1 in Figure 7. The gamma precipitates are in a rounded or cubic shape at the proper size. The effect of primary precipitate aging at 1055°C for 1 hour results in more uniform precipitation of very fine gamma prime including higher volume fraction after secondary aging comparing to the heat-treated microstructure of program No.2, which has a lower amount of primary precipitate aging at 925°C. It should be noted that such a higher temperature of primary aging might not create the early precipitation of γ' as occurring in the case of the lower temperature. Therefore, the coarser γ' precipitates do not appear after secondary aging as in program No. 2. The sufficient high temperature of primary aging at 1055°C could be considered as a carbide stabilization heat treatment. Such high temperature cannot only optimize the size and morphology of γ' but also the decomposition of the coarse, as-cast MC carbides, into fine grain boundary carbides.

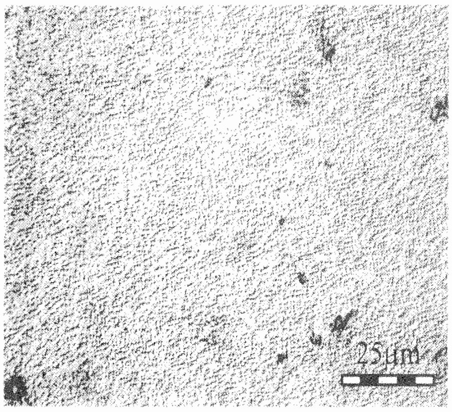

Figure 9 After heat-treatment at 1125°C/ 2hr. (AC), 1055°C/ 1 hr. (AC), and 845°C/ 24 hr. (AC).

Furthermore, after secondary aging at 845°C for 24 hours then air-cooling, gamma prime precipitates are very stable and growing very slowly. This final microstructure is expected to have better characteristics for short-term mechanical properties at elevated temperatures than those of specimens according to program No.1 and No.2. The finer γ' produced in the secondary aging treatment is good for tensile strength as well as for rupture life according to the previous works (Daleo, *et al.* 2002; and Tillack, *et al.* 1991). Especially, under creep conditions, the stable particles will become rafting or coarsening very slowly resulting in a longer lifetime. Double aging treatment is not only used commonly to control the size distribution of γ' but also to control grain boundary morphology.

According to the results of program No.4, No.5 and No.6, it should be noted that the higher solution annealing temperature could influence significantly the final microstructures in each condition, see Figures 10-12. In all cases, it was found that microstructures are very non-homogeneous consisting of coarser γ' precipitate dispersion and contain lower volume fractions of gamma prime precipitates than those of programs No.1-No.3. It should be noted that at higher solutioning temperatures of 1175°C for 2 hours, γ' forming elements would be dissolved more into the matrix than solutioning at lower temperatures of 1125°C for 2 hours, resulting in the precipitation of coarser γ' particles during aging. Moreover, it can be also seen that some areas, which are close to grain boundaries are without any gamma prime precipitates. Especially, elements needed to form these γ' could combine with carbon to be carbides and/or enter adjacent carbides being more stable carbides and/or be dissolved more into grain

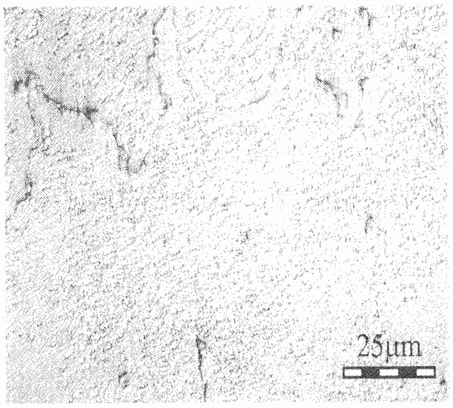

Figure 10 After heat-treatment at 1175°C/ 2hr. (AC) and 845°C/ 24 hr. (AC).

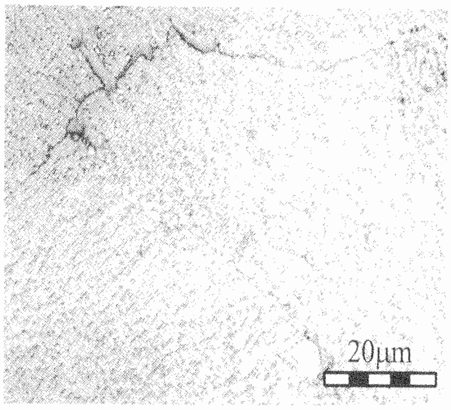

Figure 11 After heat-treatment at 1175°C/ 2hr. (AC), 925°C/ 1 hr. (AC), and 845°C/ 24 hr. (AC).

boundaries compared to the microstructures (programs No.1-No.3) with a lower solution annealing temperature. Addition of temperature 1055°C aging creates more regularly shaped particles and fine background of γ' precipitation.

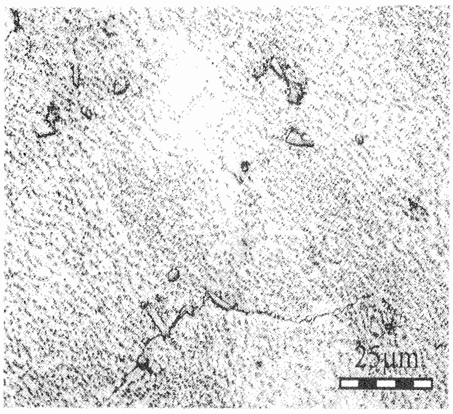

Figure 12 After heat-treatment at 1175°C/ 2hr. (AC), 1055°C/ 1 hr. (AC) and 845°C/ 24 hr. (AC).

Micro hardness tests

Table 4 Micro hardness tests.

Specimen from program No.	Average Micro Hardness (HV)	Minimum Micro Hardness (HV)	Maximum Micro Hardness (HV)
As-received material	411	405.91	416.09
1	428.4	425.12	431.68
2	408.4	401.64	415.16
3	412.8	403	422.6
4	433	427.96	438.04
5	428	424.6	431.7
6	426	423.2	428.8

From Table 4 and Figure 13, it was found that the effect of heat treatment conditions do not provide any significant difference of the results in micro hardness values. The average micro hardness values of individual heat treatment conditions are in the range of about 410-430 HV, which are not greatly different from non-reheat treated alloy. From the previous knowledge, it is well known that the hardness usually is increased with γ' particle size. However, according to the results of micro hardness tests and all microstructures of re-heat treatment conditions, Figure 6 to Figure 13, it was shown that the micro hardness results do not follow the above knowledge. This might be due to the hardness being not dependent only on γ' particle size but also shape and volume fraction of γ' particles including carbide morphology. Therefore, concerning many effects involved, it is very difficult to make a clear analysis and find the relationship only between hardness and re-heat treatment conditions due to the complex microstructure of each re-heat treatment condition.

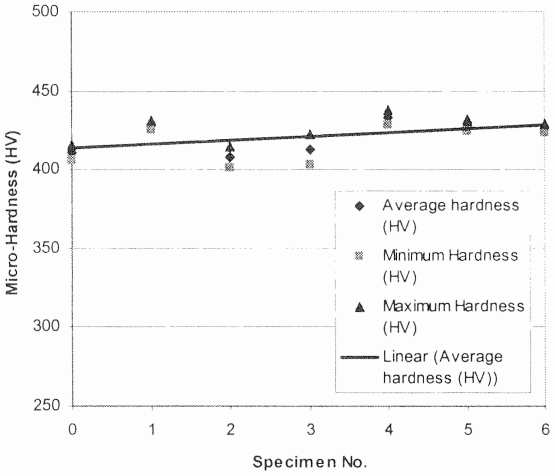

Figure 13 Micro hardness results of each re-heat treatment condition.

However, it can be summarized from Figure 13 that the maximum micro hardness values are obtained in samples from program No.1 and 4, which were reheated with simple solution and aging treatments. When adding primary aging at both temperatures (925°C and 1055°C) between solutioning at lower temperature (1125°C) and secondary aging in samples from program No. 2 and 3, their micro hardness values are similar to that of long-term service materials without any re-heat treatment. For samples from program No. 5 and 6 with a higher solutioning temperature of 1175°C, micro hardness values are a little higher than those of samples from program No. 3 and 4.

CONCLUSION

1. After various reheat treatment conditions, the microstructures according to the program No.1 and 3 seem to be the optimized microstructures for both short-time tensile and fatigue properties at elevated temperatures as well as creep rupture properties.

2. For the other programs (No. 2 and 4-6), the microstructures with the over-aging of γ' precipitates are expected to provide only creep rupture strength in some degree unless these γ' particles tend to become rafting or coarsening earlier than the more stable γ' particles of programs No. 1 and 3.

3. The similar results from micro hardness tests cannot be significant or of major concern to consider the selection of the most proper reheat-treatment conditions.

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