

## **The Relationship Between Reheat-Treatment and Hardness Behaviour of Cast Nickel Superalloy, GTD-111**

**Panyawat WANGYAO<sup>1\*</sup>, Viyaporn KRONGTONG<sup>2,3</sup>,  
Pongsak TUENGSOOK<sup>3</sup>, Weerasak HORMKRAJAI<sup>4</sup> and Nurot PANICH<sup>1</sup>**

<sup>1</sup>*Metallurgy and Materials Science Research Institute (MMRI),  
Chulalongkorn University, Bangkok, Thailand*

<sup>2</sup>*National Metals and Materials Technology Center (MTEC), Pathumthani, Thailand*

<sup>3</sup>*Production Engineering Dept., Engineering Faculty,*

*King Mongkut's University of Technology, Thonburi (KMUTT), Thailand*

<sup>4</sup>*Electricity Generating Authority of Thailand (EGAT), Nonthaburi, Thailand*

### **Abstract**

The microstructure of cast polycrystalline nickel base superalloy, GTD-111, operated for long-term services were investigated by SEM after different re-heat-treatment conditions. It was found that the exposed specimen after long-term service could be recovered in some extent by re-solution treatment followed by two-step aging treatment. The obtained dispersed precipitation of gamma prime particles was more uniform than those of the long-term exposed microstructure. The results showed that the couple of the lower (1125°C) and/or higher (1175°C) solution annealing temperatures resulted in less total volume fraction of coarse gamma prime precipitates and less homogeneous distribution in microstructure than the medium (1150°C) one. Furthermore, the increase of inserted primary aging temperatures (925°C and 1055°C) resulted in an increase of gamma prime particle size, especially of coarse gamma prime particles. The choice of re-heat treatment conditions with solutioning at 1150°C for 2 hours followed by primary aging at 1055°C for 1 hour and secondary aging at 845°C for 24 hours was the most effective when comparing to other re-heat treatment conditions in this study to optimize microstructure. Concerning the hardness test results, the hardness properties depended on the total volume fraction of gamma prime precipitated phase in case of specimens after solutioning and/or primary aging. However, in case of specimens after secondary aging, the hardness behavior trends to vary with the volume fraction of very fine gamma prime precipitated phase.

**Keywords :** Superalloy GTD-111, Microstructural Refurbishment, Re-heat-Treatment, Hardness, Superalloys, Lifetime Extension,

### **Introduction**

The Nickel base superalloy GTD-111 was designed in the 1970s and in service since 1980s in high power industrial gas turbines as the first stages blades. The alloy substituted the previous material, IN-738 LC in high power gas turbines operating at higher temperatures due to its excellent high temperature properties. The alloyed refractory elements such as Mo, W, Ta, Cr and Co help to protect the local hot corrosion. The alloy is a modification of Rene 80 and In-738 LC.<sup>(1)</sup>

The GTD-111 is a  $\gamma'$  precipitation-strengthened nickel base superalloy which widely used as turbine blades in hot sections of gas turbine engines due to its outstanding creep strength properties at high temperature as well as excellent hot corrosion resistance. GTD-111 contains a substantial amount of Al and Ti together more than 6 wt%, which provides precipitation strengthening of ordered  $L1_2$  intermetallic  $Ni_3(Al, Ti)$   $\gamma'$  phase. The size distribution and the morphology of  $\gamma'$  precipitate are affected by applied aging temperature.

However, to continue safe service it requires a repair process providing the re-establishment of the initial properties supporting the idea of refurbishment of the long-term used and exposed parts for the economic reasons. The heat-treatment processes for nickel-base superalloys continue to change in order to optimize for numerous mechanical and physical properties.<sup>(2-9)</sup> This makes allows the selection of heat treatment parameters increasingly challenging. The heat treatment processes for precipitation-strengthened nickel base superalloys are sophisticated even though they have been developed for some decades ago. The heat-treatment of nickel-base superalloys can involve solution cycles, stabilizing cycles, and aging cycles. Each thermal cycle was developed and designed for very specific metallurgical structure operation and control. Since mechanical properties are related to the microstructures. Therefore, many previous research works<sup>(10-15)</sup> have been carried out to investigate these relationships of microstructure-mechanical properties.

## Materials and Experimental Procedures

The cast nickel base superalloy in this study was GTD-111, with the following composition (wt%) 13.5%Cr, 9.5%Co, 4.75%Ti, 3.3%Al, 3.8%W, 1.53%Mo, 2.7%Ta, 0.09%C, 0.23%Fe, and 0.01%B and balance nickel. The rectangular plates about 1 cm<sup>2</sup> were cut from the most severe degraded zone of turbine blades. The plates were performed under different heat treatment conditions including solution treatment, primary and secondary precipitate aging treatments in vacuum furnace according to the experimental heat-treatment details given in Table 1. The heat-treated plates were cross-sectioned in order to study microstructure comparing to those of parallel grinded and polished surface of turbine blades. All sectioned samples were polished using standard metallographic techniques and were subsequently etched in Marble etchant, which its chemical composition is as follows 10 g. CuSO<sub>4</sub>, 50 ml HCl, and 50 ml H<sub>2</sub>O. The microstructures of heat-treated samples were observed by a scanning electron microscope with a secondary electron mode and Image Analyzer. Finally, the hardness tests were carried out in all heat-treated samples.

**Table 1.** Heat treatment conditions applied to long term exposed GTD-111

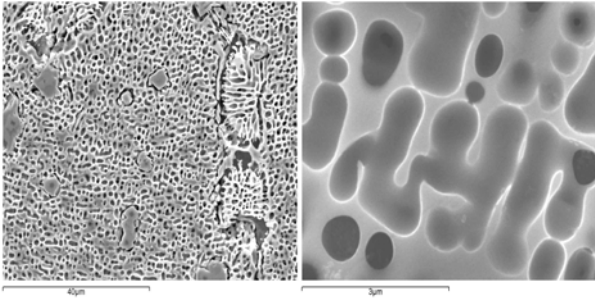
No.	Solution Treatment	Primary precipitate aging	Secondary precipitate aging
1 *	1125°C/2 hrs (AC)	-	845°C/24 hrs. (AC)
2	1125°C/2 hrs (AC)	925°C/1 hr. (AC)	845°C/24 hrs. (AC)
3	1125°C/2 hrs (AC)	1055°C/1 hr. (AC)	845°C/24 hrs. (AC)
4	1150°C/2 hrs (AC)	-	845°C/24 hrs. (AC)
5	1150°C/2 hrs (AC)	925°C/1 hr. (AC)	845°C/24 hrs. (AC)
6	1150°C/2 hrs (AC)	1055°C/1 hr. (AC)	845°C/24 hrs. (AC)
7	1175°C/2 hrs (AC)	-	845°C/24 hrs. (AC)
8	1175°C/2 hrs (AC)	925°C/1 hr. (AC)	845°C/24 hrs. (AC)
9	1175°C/2 hrs (AC)	1055°C/1 hr. (AC)	845°C/24 hrs. (AC)

\* *Standard Heat-Treatment condition*

## Experimental Results and Discussion:

### The microstructure of as-received alloy

Optical micrographs, obtained from the transverse sections at about mid blade height of the airfoil, are shown in Figure 1. The microstructure of as-cast alloy generally consists of extensive precipitation of an ordered L12  $\gamma'$  intermetallic phase within a dendrite core and in the interdendritic region. Carbides / carbonitrides predominantly of MC type, borides, sulphur-carbides and  $\gamma$ - $\gamma'$  eutectic which form during component solidification are found in smaller volume fractions locating along the interdendritic region as well.<sup>(16)</sup> Microsegregation during ingot solidification causes the formation of non-equilibrium  $\gamma$ - $\gamma'$  eutectic. The chromium carbide (M<sub>23</sub>C<sub>6</sub>) and agglomerated gamma prime particles can be seen. Coalescence of the primary gamma prime particles, as a result of long-term service, seems to occur resulting in larger and rounded particles. It was also reported that eutectic gamma prime islands as well as elongated gamma prime partials were observed as well in the work<sup>(17-18)</sup>. This is most probably due to the slow cooling rates.



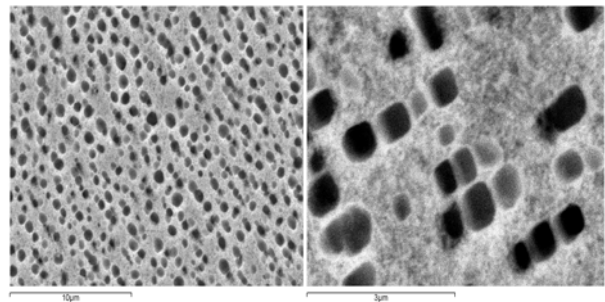
**Figure 1.** SEM micrographs of as-received microstructure after long-term service showing the coalescence of  $\gamma'$  particles, areas of  $\gamma$ - $\gamma'$  eutectic and grain boundary carbides

The degree of degradation, as evaluated by the gamma prime particle size, increases with exposed time and service temperature. The primary gamma prime particle size varied from 1.0 to 1.4 micron, as reported by.<sup>(19)</sup> In this study, however, the coarse gamma prime particle size was about 1.2 micron. The airfoil microstructure shows significant degradation in service. The primary gamma prime particles have been spheroidized and secondary gamma prime coarsened in the airfoil samples. This type of microstructure is theoretically expected to have low efficiency to hinder dislocation movements during loading at high temperatures resulting in lower creep resistance. In order to re-new the properties of the alloy, it is needed to recover microstructure to the same and/or similar to the original one by simple re-heat-treatment processes.

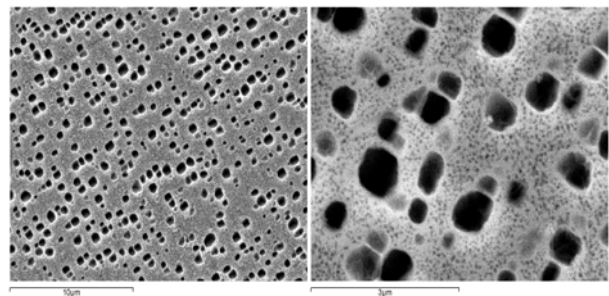
### The microstructure of heat-treated alloy

According to the previous works.<sup>(17, 20-21)</sup> repeating the standard heat treatment sequence does not work well. The structure and properties were not fully recovered by this refurbishment treatment applied to these GTD-111 blades. 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), was applied to long-term exposed specimen in this study, the more homogeneous microstructure consisting of uniform distribution of gamma prime precipitates was obtained. The size of  $\gamma'$  precipitate particles is uniform and similar in size and shape but the volume fraction of gamma prime particles is less than that of after serviced one, Figure 2. According to the heat treatment of program No.2.

(Figure 3), it was observed that the morphology is very similar to that of the standard heat-treated one, which consists of bimodal morphology. Primary precipitate aging at 925°C for 1 hour resulted in an early precipitation of very fine gamma prime  $\gamma'$  particles, which its total volume fraction is higher than that of the microstructure according to program No.1. After secondary precipitate aging, elements needed to form  $\gamma'$  precipitate would diffuse more into the former gamma prime precipitates causing the coarsening of these very fine precipitates during secondary aging. However, most of coarse irregular  $\gamma'$  particles did not further grow up during secondary aging at 845°C. Such kind of microstructure could probably provide good rupture and/or creep resistance to some degree. As it is already well known that creep strength of alloys by  $\gamma'$  precipitation is a function of  $\gamma'$  particle size. It can be concluded that a temperature of 925°C as primary aging produced more uniform and coarser of very fine rounded- $\gamma'$  particles.

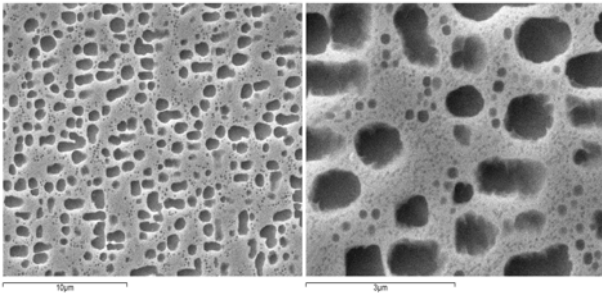


**Figure 2.** SEM micrographs of microstructures after standard heat-treatment at 1125°C/2hr.(AC) and 845°C / 24 hr. (AC); Condition No. 1



**Figure 3.** SEM micrographs of microstructures after heat-treatment at 1125°C/ 2hr. (AC), 950°C / 1 hr. (AC), and 845°C / 24 hr. (AC); Condition No. 2

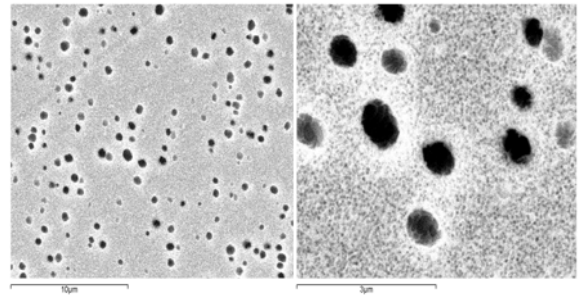
In Figure 4, the microstructures after heat treatment according to program No.3 show the more uniform dispersion of both coarse and very fine gamma prime precipitates. The microstructure is similar to that of sample No. 2 in Figure 3. The coarse gamma precipitates are in rounded or cubic shape at the proper size. The lower total volume fraction of  $\gamma'$ -phase after secondary aging comparing to the heat-treated microstructure of programs No. 1 and 2, which provided a higher amount of both primary and secondary precipitated particles, which were aged at 925°C. The  $\gamma'$  particles produced in the secondary aging treatment is good for rupture life according to the previous works.<sup>(22)</sup> Especially, under creep conditions, the stable particles would become rafting or coarsening slowly resulting in a longer lifetime. Double aging treatment is not only used commonly to control the size distribution of  $\gamma'$  particles but also to control the grain boundary morphology of carbides.



**Figure 4.** SEM micrographs of microstructures after heat-treatment at 1125°C / 2hr. (AC), 1055°C/1 hr. (AC), and 845°C/ 24 hr. (AC); Condition No. 3

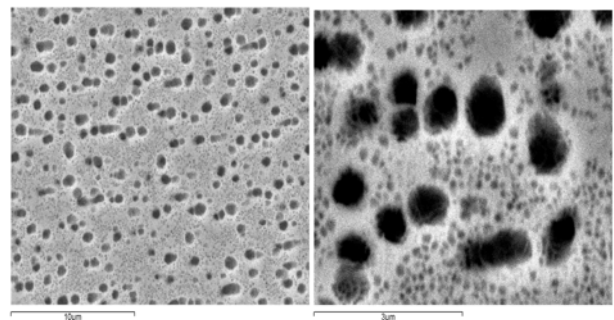
Figure 5 shows the heat - treated microstructure according to program No. 4 with higher solutioning temperature at 1150°C. The effect of this higher solutioning temperature was obviously strong on final microstructure comparing to the microstructure of program No. 1. The obtained microstructure consists of lower volume fraction of coarse gamma prime particles. The very fine  $\gamma'$  particles were also observed. It can be noted that the higher temperature of solutioning leads to more solutioning of residual coarse gamma prime

particles resulting in the smaller size and lower density of coarse  $\gamma'$  particles.

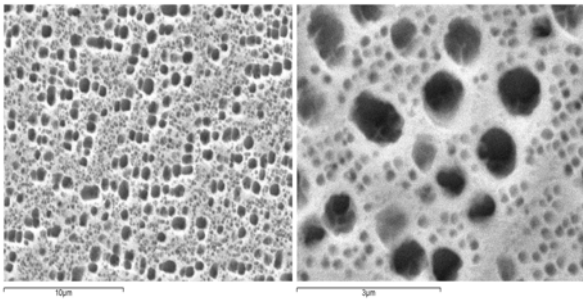


**Figure 5.** SEM micrographs of microstructures after heat-treatment at 1150°C / 2hr. (AC), and 845°C/ 24 hr. (AC); Condition No. 4

Figures 6 and 7 shows the effect of primary aging inserted between solutioning and secondary aging, on final microstructures. The inserted primary aging provided the additional precipitation of very fine gamma prime particles. The high temperature of primary aging resulted in higher volume fraction of gamma prime precipitated of both coarse and very fine  $\gamma'$  particles as Figures 6 and 7 (right) show. The obtained heat-treated microstructure according to program No. 6 might be the most proper one for both creep and tensile strengths due to its uniform distribution of both coarse and very fine  $\gamma'$  particles with the highest volume fraction when compared to the heat-treated microstructure of other programs.

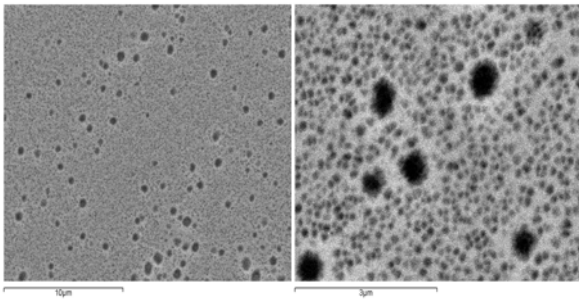


**Figure 6.** SEM micrographs of microstructures after heat-treatment at 1150°C/ 2hr. (AC), 925°C / 1 hr. (AC), and 845°C / 24 hr. (AC) ; Condition No. 5

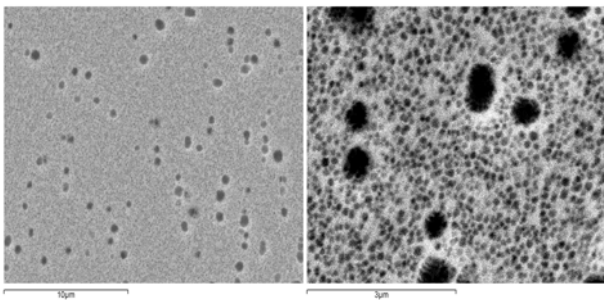


**Figure 7.** SEM micrographs of microstructures after heat-treatment at 1150°C/2hr. (AC), 1055°C / 1 hr. (AC), and 845°C / 24 hr. (AC); Condition No. 6

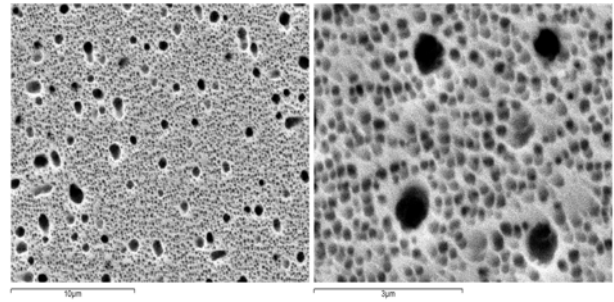
Figures 8-10 show the effect of the highest solutioning temperature on final microstructures. This highest solutioning temperature provided the microstructure with the most solutioning of residual coarse  $\gamma'$  phase particles resulting in very low volume fraction of coarse  $\gamma'$  precipitated particles. The inserted primary aging did not influence on the size, distribution and volume fraction of coarse  $\gamma'$  particles but do greatly on the very fine one.



**Figure 8.** SEM micrographs of microstructures after heat-treatment at 1175°C/ 2hr. (AC) and 845°C/ 24 hr. (AC); Condition No. 7



**Figure 9.** SEM micrographs of microstructures after heat-treatment at 1175°C/2hr. (AC), 925°C/ 1 hr. (AC), and 845°C/ 24 hr. (AC); Condition No. 8

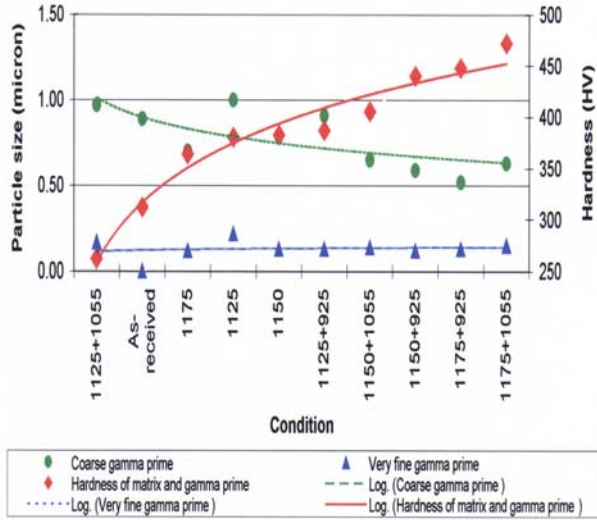


**Figure 10.** SEM micrographs of microstructures after heat-treatment at 1175°C/ 2hr. (AC), 1055°C/ 1 hr. (AC), and 845°C/ 24 hr. (AC); Condition No. 9

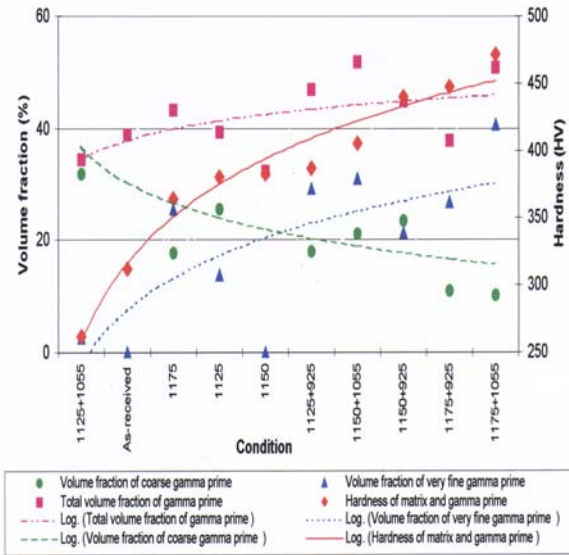
### Hardness Tests

The summary of the obtained results concerning the particle size and volume fraction for all reheat-treatment conditions are illustrated Figures 11-14. The Figure 11 shows the effect of gamma prime particles size on the hardness property of specimens under each heat treatment condition before final aging. It is clearly seen that the size of very fine gamma prime particles has no significant effect on hardness properties. However, in contrast, the size reduction of coarse gamma prime particles (resulting in an increase of volume fraction of very fine gamma prime precipitation) is probably related to the increase of hardness. The minimum hardness that was obtained from the heat treatment is as follows : solutioning at 1125°C for 2 hr. and primary aging at 1055°C for 1 hr., which microstructure consists of many coarse gamma prime particles and low content of very fine gamma prime particles. The maximum hardness is obtained from the heat treatment consisting of the highest solutioning temperature in this study. This heat treatment condition is as follows : solutioning at 1175°C for 2 hr. and primary aging at 1055°C for 1 hr. providing the very high content of very fine gamma prime particles and low fraction of coarse gamma prime particles. Figure 12 shows that the hardness increases with the volume fraction of very fine gamma prime particles and total volume fraction of gamma prime particles of heat-treated specimens before final aging. However, the hardness is more related to the volume fraction of very fine gamma prime particles rather than to the latter. Furthermore, the increase of volume fraction of more precipitated very fine gamma prime particles is also reasonably linked to the decrease of volume fraction of precipitated coarse gamma prime particles. Figures

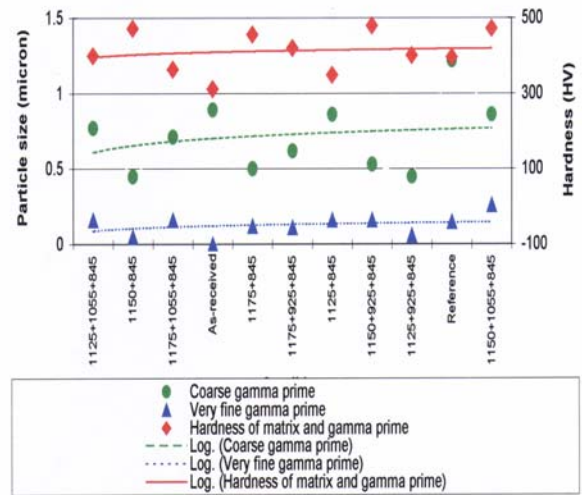
13 and 14 show the approximated trends that the hardness slightly increases as the slow increasing of particle size and volume fraction of all specimens after final aging.



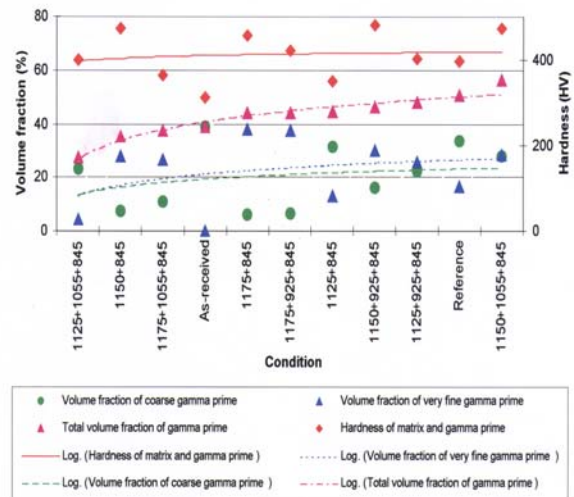
**Figure 11.** The relationship between size of gamma prime particles and hardness in each heat treatment condition before final aging (From minimum to maximum hardness)



**Figure 12.** The relationship between hardness and total volume fraction (Before final aging) of gamma prime phase



**Figure 13.** The relationship between size of gamma prime particles and hardness in each heat treatment condition after final aging (From minimum to maximum hardness)



**Figure 14.** The relationship between hardness and total volume fraction (After final aging) of gamma prime phase

**Conclusion**

1. After various reheat treatment conditions, the microstructures according to the program No.6 seems to be the optimized microstructures for mechanical properties at elevated temperatures due to its maximum volume fraction.

2. The higher temperature of solutioning provided the greater solutioning of coarse  $\gamma'$  particles in smaller size. However, the increase of inserted primary aging temperature resulted in an increase of gamma prime particle size.

3. To re-precipitate the gamma prime, it is necessary to solution the elements that needed to precipitate later. Therefore, it is very important to have enough elements (Al, Ti, Ta and Nb) in matrix for next precipitation.

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