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Investigation of the Heat Treatment Effect on Microstructures and Phases of Inconel 713C Superalloy

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ABSTRACT

The application of Inconel 713C is very vast in different industries such as in automobiles, aircrafts, boilers and turbines. In this paper, Inconel 713C specimens were solutionized at 1000 and 1200 °C for 1 hours in order to study changes in phases and the structure of alloy to modify the γ' phase. The optical and the scanning electron microscopy were used to investigate the structure and the energy dispersive spectroscopic (EDS) and the X-ray diffraction (XRD) were applied to identify the composition and phase changes. Results showed that the solutioning heat treatment at 1000 °C for 1 hour caused to participate $Cr_{23}C_6$, but when the temperature of heat treatment increased to 1200°C, chromium carbides solutionized in the γ matrix. Besides, by applying the heat treatment at temperature of 1000 °C, the γ' phase dissolved in (200) and (220) plane and this phase in the plane of (111) had the best stability. However the (220) plane of γ' phase was stable thermodynamically for the specimen without the heat treatment. Phases of Ni_5Al_3 and $Al_{0.42}Ni_{0.58}$ were observed at the temperatures of 1000 and 1200 °C, respectively. In addition, as the heat treatment temperature increased from 1000 to 1200 °C the new phase of $AlNi_3C_{0.5}$ solutionized in the γ matrix.

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1. INTRODUCTION

Inconel 713C (IN 713C) belongs to the group of vacuum melted Ni-based superalloys. It was developed in the 1950s, and due to its extraordinary properties, it was widely used in different high temperature applications, even as a blade material in the turbocharger blades or turbines [1, 2]. As, IN 713C can tolerate harsh operating conditions (high temperatures, temperature gradients, abrupt thermal changes, oxidizing and corroding atmosphere, high pressures and multiaxial stresses), it is used in different industries such as automobile, nuclear [3], petrochemical [4] and aerospace industries [5, 6]. This superalloy contains aluminum and chromium to form the protective impermeable Al_2O_3 and Cr_2O_3 oxide films that supply the corrosion resistance for high temperature application [7]. Thus, it is considered to be a superior material in comparison to other materials such as aluminum–magnesium alloys and maraging steels due to its high resistance to corrosion and good strength at high temperature [8].

Inconel superalloys are manufactured through the casting, forging and heat treatment process. The microstructure changes induced during the manufacturing process affects the performance of the alloy during service [7]. Several studies [1, 9-11] showed that the microstructure of precision cast Inconel superalloys parts consisted of dendritic γ-grains, primary and eutectic MC carbide, and coherent γ' precipitates with a cubic (L12) crystal structure. High solubility of many elements such as cobalt, iron, chromium, molybdenum, titanium and tungsten gives the possibility of strength of austenitic matrix γ [12, 13]. Phase γ' takes a high volume compared to its mass and is the result of the strong attractive bonds between various atoms and because of that it has specific mechanical and physical properties [13, 14]. Two types of carbide with NaCl-type structure are observed: the

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normal one (MC) with lattice constant a= 0.40 nm and an anomalous one ($M_{23}C_6$ and MC_6) with a=0.43 nm. The latter carbide contains some Zr. Sometimes the γ/γ' eutectic and MC carbide are also observed in the Inconel group microstructure [1, 9, 13, 15].

The heat treatment of superalloy is an important step for achieving an optimum microstructure and mechanical properties such as hardness, fatigue and creep behavior [15-17]. Both solid solution and precipitation strengthening are the major steps in strengthening mechanisms [18]. Although a number of studies have focused on the structure and different phases of Inconel 718 [10, 11, 18-20], a few reports dealt with the structure and different phases of IN 713C.

Most reports about the IN 713C properties were published by Binczyk et al. [21-25]. They studied about the macro-structure of IN 713C superalloy after the γ' volume modification in a stepped test casting method. They used different solification processes and then investigatd the effects of columnar and equiaxial crystals on mecanical properties. They concluded that the thermal analysis offered more possibilities for the interpretation of the first stage of the solidification process.

Thus, in this paper through the solutioning heat treatment process at two different tempratures as variables, the IN 713C specimens were studied in order to modify the γ' phase and the solid solution strengthening effect. Besides, we used different chemical etchants to study the microstructure of IN 713C since no particular etchant has been introduced for Inconel 713 C until now. Then we used the optical microscopy (OM) and the scanning electron microscopy (SEM) to detect the structure and used the XRD and the EDS for detecting the existing phases in various specimens.

2. MATERIALS AND METHODS

The used material was IN 713C nickel superalloy with the chemical composition as shown in Table 1 that was balanced with nickle and compared with the ASM standard references [26].

The alloy specimens were in the form of cubic shape with dimensions of 10×10×10 mm. Specimens preparation for studying the alloy structure was carried out using the standard mechanical grinding and polishing techniques. The specimens for optical and electron microscopy were etched by immersing in different chemical solutions. Details about the chemical composition of the corrosive solutions are provided in Table 2.

For changing the structure and phases, the solutioning heat treatment was done. The heat treatment program involved a solution treatment at two different temperatures (1000 °C and 1200 °C) in 1 hour followed by the air cooling. The XRD was performed using an X-ray powder diffractometer, Philips PW 1710 (Cu Ka radiation, 25°C). The curves were recorded at a scanning rate of 0.0258/s with 2Ɵ (Bragg angle) scan range from 10 to 90 degree. The SEM was equipped with energy dispersive x-ray spectroscopy (EDS). For the imaging and EDS analyses, a 20 kV accelerating voltage was used.

TABLE 1. Chemical composition of used samples

Element	Al	Ti	Ta+Nb	Zr
Standard	5.50-0.50	0.50-1.00	1.80-2.80	0.05-0.15
Sample	5.50	0.97	1.905	0.06
Element	**Mn**	**Cr**	**Mo**	**Si**
Standard	Max 0.25	12.00-14	3.80-5.20	Max 0.5
Sample	0.04	14.00	4.50	0.04
Element	**C**	**Fe**	**B**	**Cu**
Standard	0.08-0.20	Max 2.50	0.005-0.015	Max 0.5
Sample	0.12	0.13	0.006	0.005

TABLE 2. Chemical composition of used etchants

Etchant number	Composition
No. 1-new	HCl (5 ml)+ HNO_3(1 ml)+ ethanol (6 ml)
No. 2	HCl (5 ml)+ HNO_3(1 ml)+ glycerine (6 ml)
No. 3-marble	HCl 37% (50 ml) + $CuSO_4$ (10 g) + H_2O(50 ml)
No. 4	1% HF+33% HNO_3+33% Acetic Acid+33%H_2O

3. RESULTS AND DISSCUSSION

Figure 1 shows the typical OM images of the IN 713C superalloy microstructures with different etchants for the specimen without the solutining heat treatment (WHT or as-cast) with magnification of 50x.

In Figure 1 (a) the dendritic shape of carbides is depicted and carbides are observed in a continuous shape. With increasing the alcoholicity of the chemical etchant (the glycerin was replaced by ethanol in etchant No.2) pores were also observed in Figure 1 (b). As the marble etchant was a strong chemical solution, details of phases could not be seen in this magnification, although the etching time was low (about 1 seconds). (Figure 1 (c)). Some areas of the specimen surface seemed to be etched extremely and observed in the darker color. As seen in Figure 1 (d), grain boundaries could be detected obviously when the etchant No. 4 was used. Three assumed grains were drawn in Figure 1 (d). In addition, the carbide prominences were not shown. In this way, the grain size could be measured about 150 μm under OM images. Thus, the etchant No. 4 could be suggested when the the grain size was tended to be measured.

Figure 2 depicts the morphology of γ'- Ni_3Al phase in the γ phase matrix (nickel-based solid solution) and alloy carbides for IN 713C with the magnification of 200x when three etchants were used for evaluting different phases. It is noticeable that the casting alloy of IN 713C was one of the first developed nickel-based superalloys hardened by intermetallic phase γ'-Ni_3Al. The intermetallic phase content also exceeded more than 50% [27].

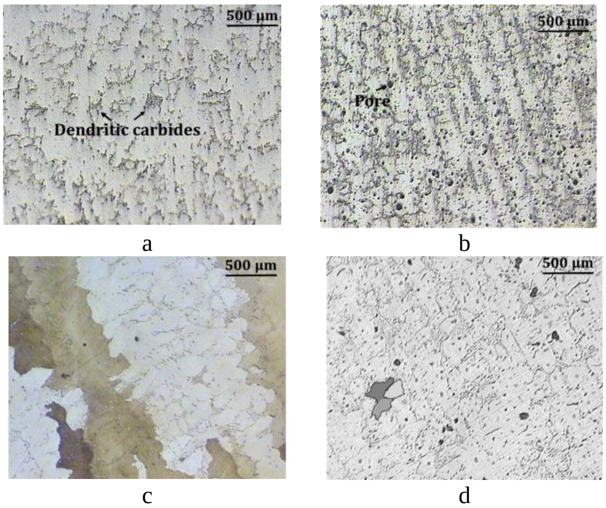


Figure 1. Comparison of optical microscopic images of IN 713C with various etchants– magnification 50x: a) No.1, b) No.2, c) No.3 and d) No.4

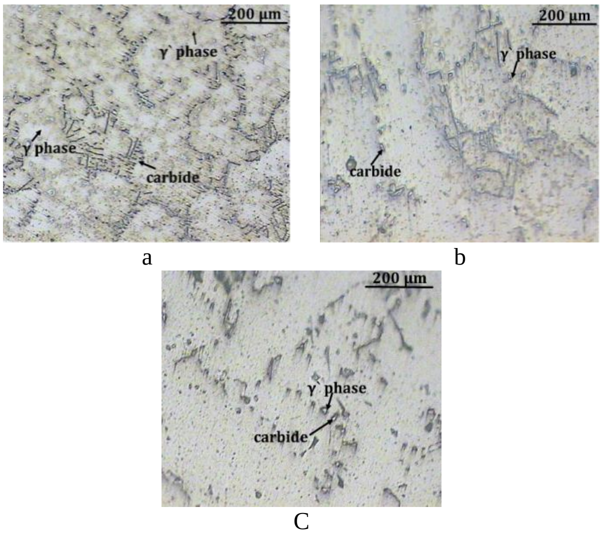


Figure 2. Optical microscopic images of IN 713C with different etchants-magnification 200x with various etchants: a) No. 1, b) No. 2 and c) No. 3

As shown in Figure 2 (a), the shape of alloy carbides such as chromium, niobium, aluminum-nickel carbides (due to EDS results in Figure 7 or XRD results in Figure 8) was irregular at grain boundaries when the new etchant (No.1) was used. Thus, appling such etchant could be suggested when the morphology of carbides was improtant to study. Grain boundaries were faded in Figure 2 (b) and (c) since other etchants were used. Figure 2 (b) and (c) show that in some areas the γ'-Ni_3Al phase surrounded alloy carbides. The shape of γ'-Ni_3Al phase seemed to be spherical. As Nguyen et al. [28] claimed, under normal conditions, the magnitude of the lattice misfit determined the shape of the γ' precipitates, which could change from spherical (for misfits in the range 0-0.2%), to cuboidal (0.5-1%), and plate-like (above 1.25%) [28]. Due to this claim, the lattice misfit was about 0-0.2% as the shape of γ' precipitates were spherical. Bhambri et al. [9] also claimed that under planar front growth conditions the carbide particles were octahedral, under cellular growth conditions and they were plate like, elongated along the cellular growth direction. Under dendritic growth conditions they had irregular shape. They also showed that the nature of carbide phases that are desirable in polycrystalline nickel-base superalloys for high temperature grain boundary strengthening depended on the composition of the superalloy. The morphology of carbides could also change from blocky, rod-like into script-like due to the increasing carbon contain [29]. According to other research results [29], the irregular shape of carbides resulted from the dendritic growth condition.

OM images of IN 713C with magnification of 500x with etchant No.3 for different specimens without and with heat treatment are shown in Figure 3 (a), (b) and (c). When the IN 713C heated from 1000 to 1200 °C, some of the γ' phase and alloy carbides dissolved in the matrix and the residual carbide size decreased to at least 20 micrometers as shown in Figure 3. The solutiong heat tratment also concluded to lower γ' phase. Figure 3 (d), (e) and (f) also show the grain size of different specimens. The solutioning heat treatment led to the increase of the grain size from 170 to 200 μm, when the solutioning temperature was increased from 1000 to 1200 °C. The heating caused the increase of the grain size of the γ matrix to enhance the mechanical properties. Such behavior was also reported in the liturature [17]. Li et al. [17] reported that the change in the morphology and size of the γ' precipitates was atributed to the high temptrature up to 1100 °C.

The XRD result also is depicted in Figure 4. Thus phases in IN 713C without heat treatment were: 1) ordered face center cubic (FCC) γ'-Ni_3Al precipitates with a=3.572 A°. This phase is coherently precipitated in a γ-matrix [17], 2) alloys carbides such as NbC and $AlNi_3C_{0.5}$ with a=4.4 A° and a=3.62 A°, respectively.

The most stable plane of γ' phase in this specimen was (200). Such behavior was also reported for other types of Inconels [4].

When the sample heat treated at 1000 °C, few Cr-rich $M_{23}C_6$ secondary carbides plus NbC carbide were observed within grains and in some grain boundaries. This observation was consistent with the previous results [3].

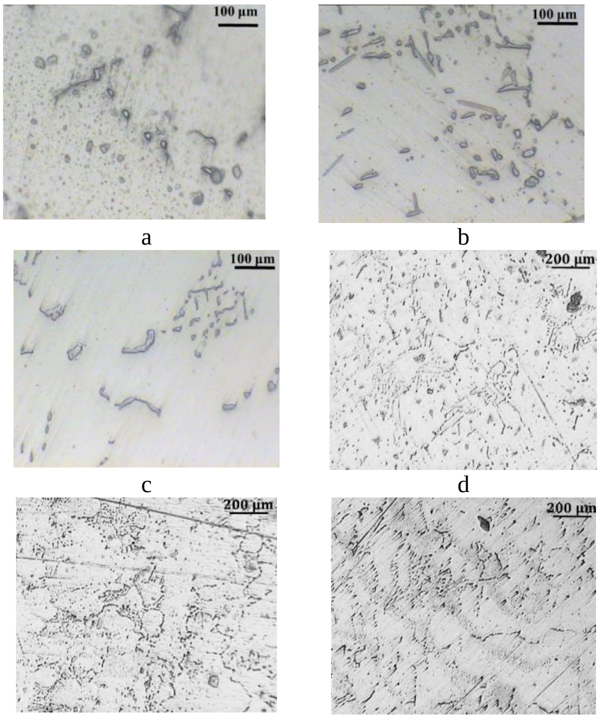


Figure 3. Optical microscopic images of IN 713C with etchant No.3: a) without heat treatment, b) heat treated at 1000 °C and c) heat treated at 1200 °C and with etchant No.4, d) without heat treatment, e) heat treated at 1000 °C and f) heat treated at 1200 °C

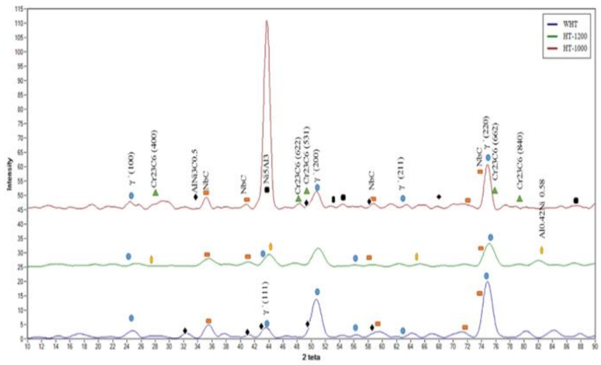


Figure 4. XRD results for specimens a) without heat treatment: blue line, b) heat treated at 1200 °C: green line and c) heat treated at 1000 °C: red line efficiency on a single square in channel

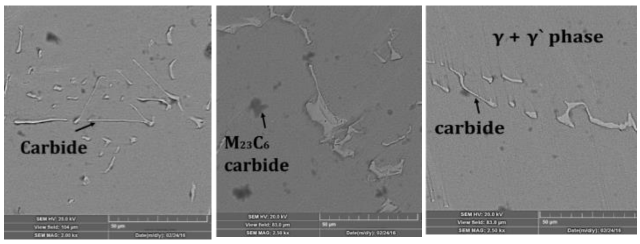
Mankins et al. [30] and Wu et al. [31] confirmed for Alloy 617 that the γ' phase solves temperature (871 °C) was lower than the $M_{23}C_6$ solves temperature (much higher than1093 °C). Some MC carbide were found to be completely unstable between 925 to 1147 °C, forming $M_{23}C_6$ due to the following reaction [9]:

$MC + \gamma = M_{23}C_6 + \gamma'$

But among MC type carbides, TaC and NbC were very stable [9]. The phase of Ni_5Al_3 with the orthorhombic crystal structure (a=7.475, b=6.727 and c=3.732 A°) also appeared at temperature of 1000 °C. There was no M_6C carbide in any samples although other researches claimed that M_6C carbide was identified in as-cast IN 713C [9]. The γ' phase solved in the plane of (200) and (220) but the peak height increased in the (111) plane due to the above reaction. As shown in XRD results, with solutioning heat treatment at 1200 °C, the height of γ' phase decreased and he most stable plane of γ' phase in such specimen was (200). Besides, another reserch [32] reported that the eutectics and coarse γ′ would be dissolved after the heat treatment with the peak temperature of 1330 °C.

In addition, the NbC carbide peaks were also seen in the XRD pattern. This observation showed that the solutioning temperature was not high enough to solve MC type carbides. However, the height of NbC decreased and such result showed the NbC were unstable at this temperature. The phase of $Al_{0.42}Ni_{0.58}$ with cubic crystal structure (a=2.87 A°) was also observed. The XRD observations showed no δ phase presented in samples as the δ phase emerged at temperatures from 750 to 1020 °C [4, 6]. When the temperature was higher than 980 °C, most δ phases with orthogonal and ordered structure dissolved [6, 33, 34]. The δ phase was an intermetallic phase frequently presented in the alloy and had the orthorhombic incoherent Ni_3Nb chemical composition [4].

SEM images of IN 713C are shown in Figure 5, where carbides were equally distributed along the matrix. Some of carbides were relatively large, which were NbC type or CrC type carbide as verified by EDS analysis.



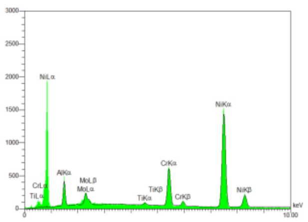
a b c

Figure 5. Scanning electron microscopic images of IN 713C with etchant No.3: a) without heat treatment, b) heat treated at 1000 °C and c) heat treated at 1200 °C

After the solutioning treatment, most of carbides were dissolved back into the matrix, however there were still some carbides left, as reported by other researches [18]. The microstructure evolution of superalloys was significantly affected by the solutioning heat treatment.

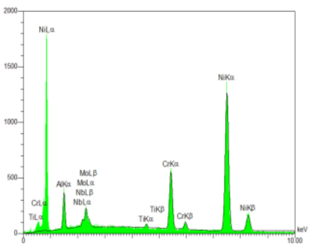
The mean size of γ'- Ni_3Al precipitates decreased with increasing heat treatment temperature to 1200°C. Some researchers have pointed that the cooling rate after the solutioning treatment could affect the size and shape of γ'-precipitates [16]. This precipitates contributed to increase the hardness [34, 35]. Besides, the alloy had at least 5% γ' volume fraction with an average precipitate size of around 5–10 µm.

Figures 6 and 7 depict EDS results for various specimens. EDS result for specimens in the area of the γ+γ' phase and carbides phase are shown in Figures 6 and 7, respectively. The chromium content decreased in the carbide phase as the heat treatment temperature increased to 1200 °C and small amount of carbon appeared in the γ matrix. This result showed that the $Cr_{23}C_6$ was dissolved in the γ matrix. Besides the titanium content increased in the solutionized heat treatment with respect to the specimen without heat treatment.



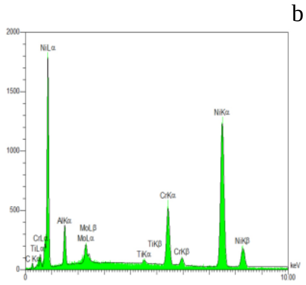
Element	%W	%A
Al	0.35	0.75
Ti	0.13	0.16
Cr	4.70	5.27
Ni	93.97	93.30
Mo	0.85	0.52

a



Element	%W	%A
Al	0.35	0.75
Ti	0.12	0.14
Cr	4.71	5.28
Ni	93.91	93.26
Mo	0.71	0.43
Nb	0.21	0.13

b



Elements	%W	%A
Al	0.33	0.72
Ti	0.15	0.18
Cr	4.59	5.12
Ni	93.89	92.75
Mo	0.90	0.54
C	0.14	0.68

c

Figure 6. EDS result for specimens in the area of the γ +γ' phase a) without heat treatment, b) heat treated at 1000 °C and c) heat treated at 1200 °C

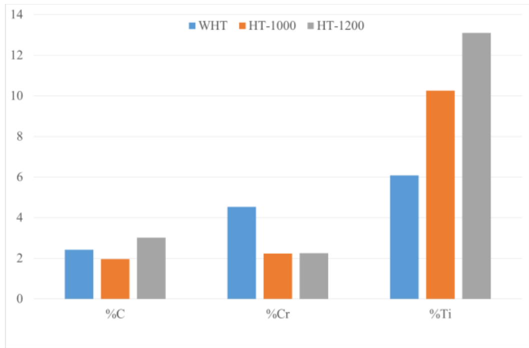


Figure 7. EDS results for specimens in the area of carbides phase a) without heat treatment: WHT, b) heat treated at 1000 °C and c) heat treated at 1200 °C

4. CONCLUSION

In this paper, the heat treatment and different types of etchants were variables for changing the phase and the micro structure of the IN 713C nickel superalloy. The heat treatment was solutioning at 1000 and 1200 °C for one hour and cooling at the ambient air. The finding of this paper could be summerize as following:

- The XRD and the EDS results showed that the heat treatment at 1000 °C caused the formation of $Cr_{23}C_6$ and participating the phase of Ni_5Al_3. However these two phases solved in the matrix when the solutioning temperature incresed to1200 °C.
- By increasing the solution temperature to 1200 °C, the phase of $Al_{0.42}Ni_{0.58}$ formed and the NbC was dissolved in the γ matrix. The most stale plane of γ' phase was (111) and similer to as- cast specimen.
- The new etchant with the mentioned chemical composition was appropriate when the carbides morphology was the subjective of the study.
- The solutining heat treatment caused increase of the grian size up to 200 µm, since the temperature was about 1200 °C. The best etchant for measuring the grain size was etchant No.4

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Investigation of the Heat Treatment Effect on Microstructures and Phases of Inconel 713C Superalloy

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چکیده

کاربرد اینکونل 713C در صنایع مختلف از جمله صنعت خودروسازی، هواپیماسازی، توربین‌ها و بویلرها گسترده شده است. در این مقاله، نمونه‌هایی از جنس اینکونل 713C در دماهای ۱۰۰۰ و ۱۲۰۰ درجه سانتی‌گراد مورد عملیات حل-سازی قرار گرفته است تا تغییرات فازی و ریزساختاری آلیاژ به منظور بهینه کردن میزان فاز گاماپرایم مورد ارزیابی قرار گیرد. میکروسکوپ نوری و الکترونی برای بررسی ریزساختار، روش طیف‌سنجی تفرق انرژی و تفرق پرتو ایکس به منظور مطالعه تغییرات فاز و ترکیب استفاده شدند. نتایج نشان می‌دهد که عملیات حل‌سازی در دمای ۱۰۰۰ درجه سانتی-گراد و زمان یک ساعت موجب رسوب‌دهی کاربید کروم ($Cr_{23}C_6$) می‌گردد، اما زمانی که دمای عملیات حرارتی به ۱۲۰۰ درجه سانتی‌گراد برسد کاربید کروم در زمینه گاما حل می‌شود. در ضمن اینکه عملیات حرارتی در دمای ۱۰۰۰ درجه سانتی‌گراد موجب می‌شود که فاز گاماپرایم در صفحات (۲۰۰) و (۲۲۰) حل شده اما در صفحه (۱۱۱) بیشترین پایداری را داشته باشد. هرچند که پایداری فاز گاماپرایم در صفحه (۲۲۰) برای نمونه بدون عملیات حرارتی از لحاظ ترمودینامیکی پایدار است. فازهای Ni_5Al_3 و $Al_{0.42}Ni_{0.58}$ به ترتیب در دمای ۱۰۰۰ و ۱۲۰۰ درجه سانتی‌گراد پایدار هستند. علاوه بر آن، زمانی که دمای عملیات حرارتی از ۱۰۰۰ به ۱۲۰۰ درجه سانتی‌گراد افزایش یابد، فاز جدید $AlNi_3C_{0.5}$ در زمینه گاما هم حل می‌شود.

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