Effects of γ' Precipitation on the Structure and Properties of 713LC Superalloy *via* HIP Treatment

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This study aimed to examine the TEM observation of γ' precipitate morphology on 713LC superalloy *via* HIP treatment, and evaluate the effects of HIP treatment on the 713LC alloy. The experimental results showed that an obvious distribution of γ' precipitations appeared in the γ matrix for 713LC superalloy after HIP treatment. It also demonstrated that the grain sizes are uniform and the porosity of the structure is improved. The strengthening mechanism of the γ' phase was Ni_3Al precipitates, and it increased the strengthened phase and mechanical properties of 713LC superalloy. Meanwhile, the porosity decreased to 60.3%, hardness increased to 40.5 HRC, and density enhanced to $7.92 \times 10^3 \text{ kg/m}^3$. Thus, this study confirmed that tensile strength and elongation of 713LC superalloy can be increased through HIP treatment.
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1. Introduction

HIP has long been used as a pressing technique to achieve full densification of sintered parts and castings, diffusion bonding and pressure impregnation. It is a process that uniquely combines high pressure and temperature to produce materials and parts with substantially better properties than those by other methods.¹⁾ Elimination of defects in superalloy casting or in large sintered cemented carbide parts is probably today's most widely applied use of HIP for metal.^{1,2)} However, until recently, hot isostatic pressed material has been used for more common applications.

The aerospace industry often uses nickel-based superalloys for the blades of jet engine turbines. Normally, nickel-base superalloys are defined as those alloys that have nickel as the major constituent, with significant amounts of chromium. They may contain cobalt, iron, molybdenum, and tantalum as major alloy additions. These alloys are strengthened by solid solution and second phase intermetallic precipitation.^{3–5)} 713LC is a cast nickel-base precipitation hardened alloy that combines superior cast ability and creep resistance. Several strengthening mechanisms take effect in this alloy, the main mechanism is precipitation strengthening by coherent precipitates of inter-metallic phase Ni_3Al or $\text{Ni}_3(\text{Al}, \text{Ti})$.^{6,7)} Owing to its excellent high temperature properties and thermal stability, the 713LC superalloy has been widely used in aviation industry.^{8–10)}

Several advanced melting and casting techniques have been developed for this superalloy, but melting related problems, such as segregation, porosity and uniform precipitation, may lead to degradation of the mechanical properties of the alloy. A major purpose of HIP is the production of dense and homogeneous material. Furthermore, it is of importance to be aware of effects that may lead to residual pores or an inhomogeneous microstructure development during HIP process. Therefore, this study explored the microstructure and characteristics of γ' precipitations for 713LC superalloy through HIP treatment. The structure

changes were studied using SEM microscopy and transmission electron microscopy (TEM). Moreover, tension tests on mechanical properties of 713LC castings were conducted, both at room temperature and high temperature. Porosity tests, hardness tests and fracture microstructure inspections were used to evaluate the effects of HIP treatment on 713LC superalloy.

2. Experimental

713LC superalloy contains neither Co nor Ta, and should therefore does not present any contamination problems. The chemical composition (mass%) of the HIP treated for 713LC cast superalloy is shown in Table 1. The alloys were prepared by VIM process (vacuum induction melting). After melting, the alloy was solidified into square plate castings. All the specimens of tension tests were obtain from the castings by WEDM (wire electrical discharge machining). The specimen sizes of the tension tests (CNS 2112 G2014) are shown in Fig. 1. For the tension tests, a SHIMADZU universal material test machine with a maximum load of 25 tons was used.

In earlier studies, Chang *et al.* studied the effects of different parameters of HIP treatments for 718 and 713LC superalloys,^{11–14)} and reported that the key parameters of the HIP process include temperature, pressure, and soaking time. Their experimental results showed that HIP treatment for 713LC alloy at 1180°C, 175 MPa, and 2 h is the optimal process. Hence, this study compared the microstructure and mechanical properties of as-heat treated and optimal HIP treatment on 713 alloy. SEM and TEM microscopy was used to observe the γ and γ' precipitations for 713LC superalloy, and the effects of γ' precipitations and distributions for 713LC superalloy *via* HIP treatment were also studied. HIP equipment was from USA Flow Pressure Systems, the commercial HIP equipment contains a Uniform Rapid Cooling (URC) system that offers a decreased cycle time, higher productivity, and combined solution heat treatment.¹⁵⁾

After HIP treatment, all the specimens were subjected to the following heat treatment: solution was treated at a

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Table 1 Chemical composition of Inconel 713LC superalloy (mass%).

Ni	Cr	Mo	Nb	Al	Ti	Fe	Mn	C	V	Cu
75.23	11.89	4.63	1.98	5.37	0.78	0.03	0.02	0.05	0.01	0.01

Fig. 1 The specimen size of tension test for 713LC superalloy.

temperature of 1175°C for 2 h, followed by N_2 cooling, aged at 650°C for 16 h, and then air cooled to room temperature.⁵⁾ The solution-treating and age-hardening processes were performed in a commercial Germanic SCHMETZ vacuum furnace. Porosity testing followed the ASTM C373-88 standard. Hardness tests were measured by HRC with loading of 150 kg, which complies with the CNS 2114 Z8003 standard. Moreover, a specific gravity test followed the ASTM D792. To evaluate the effects of γ' precipitation of 713LC by HIP process, tensile tests at room and high temperature through with two different strain rates were evaluated. The microstructure, porosity, and TEM inspections were performed.

3. Results and Discussion

Figure 2 shows the SEM micrographs of casting, as-heat treated and 1180°C, 175 MPa, 2 h HIP treatment for 713LC superalloy. The γ' precipitation was not in uniform distribution in the matrix after casting condition, as shown in Fig. 2(a). It shows the incomplete solution of the microalloy elements of the matrix on precipitation. In comparison with the volume of γ' precipitation, there are few fine and non-uniform precipitations appearing in the γ base after solid solution and aging treatment, as shown in Fig. 2(b). However, more fine grain size and uniformity of γ' precipitations were distributed in the γ matrix after HIP treatment, as shown in Fig. 2(c). Significantly, the alloying elements of precipitation were more completely. As seen, the most common phenomenon is that the γ' uniformly precipitates out first from γ matrix. HIP treatment also could increase the γ' precipitation to strengthen the effectiveness of precipitation. Since the γ' precipitation was the main strengthening phase for 713LC superalloy,¹⁶⁾ increasing the fine graining of the γ' precipitation would effectively strengthen the mechanical properties of 713LC superalloy.

Table 2 compares the hardness, properties, and specific gravity of as-heat treated and HIP treatment of 713LC superalloy. Most of the nickel-based superalloys used

Fig. 2 SEM micrographs of 713LC superalloy (a) casting, (b) as-heat treated, (c) 1180°C, 175 MPa, 2 h HIP treatment.

Table 2 Comparison the properties of as-heat treated and 1180°C, 175 MPa, 2 h HIP treatment for Inconel 713LC superalloy.

	Hardness (H/HRC)	Porosity (%)	Specific gravity (kg/m ³)
As-heat treated	39.7	0.209	7.87×10^3
HIP treatment	40.5	0.083	7.92×10^3

Table 3 Tensile properties of as-heat treated and 1180°C, 175 MPa, 2 h HIP treatment for Inconel 713LC superalloy at all the temperatures studied (strain rate is 0.001 s⁻¹).

	Temper. (T/°C)	Tensile Strength (σ/MPa)	0.2% Yield Strength (σ/MPa)	Elongation (%)
As-heat treated	25	854.51	803.59	1.67
	540	831.45	824.21	1.94
	650	898.08	883.89	1.95
HIP treatment	25	929.31	906.2	2.22
	540	939.45	918.38	2.36
	650	1020.29	1003.18	3.89

Table 4 Tensile properties of as-heat treated and 1180°C, 175 MPa, 2 h HIP treatment for Inconel 713LC superalloy at all the temperatures studied (strain rate is 0.0001 s⁻¹).

	Temper. (T/°C)	Tensile Strength (σ/MPa)	0.2% Yield Strength (σ/MPa)	Elongation (%)
As-heat treated	25	824.94	775.81	1.81
	540	878.02	853.08	2.25
HIP treatment	25	925.42	895.95	2.36
	540	967.08	934.55	2.78

vacuum melting for production, but segregation, porosity, and non-uniformity of the microstructure are problems generated during the solidification process. These defects degrade the mechanical properties of the alloys.¹⁾ Elimination of porosity *via* HIP treatment could improve fatigue and tensile properties. In this study, the HIP treatment showed that by increasing the hardness of HRC 40.5, even after tension tests at a temperature of 650°C, the hardness of HIP-ed sample slowly increased to HRC 40.7. It shows that the 713LC superalloy has better high temperature resistance after HIP treatment. In fact, the HIP treatment combination of pressure and temperature can be used to achieve a particular density at lower temperatures. The effect of the lower temperature is that unacceptable grain growth can be avoided, as the high external pressure collapses gas-filled pores and gives full density. Therefore, porosity decreased about 60.3% (0.209% → 0.083%), the density was increased to $7.92 \times 10^3 \text{ kg/m}^3$, which is helpful to enhance the mechanical properties of 713LC superalloys.

Table 3 shows the tensile strength, 0.2% yield strength, and elongation of as-heat treated and HIP treatment for 713LC superalloy at all temperatures tested in this study (strain rate is 0.001 s⁻¹). HIP densification is a multi-mechanism process, in which the dominant mechanism dominate depends on the process parameters (temperature, pressure and soaking time).^{2,11–14)} Functions of HIP processes include defect healing of castings, removal of internal porosity, improvement in property scatter and mechanical properties. Its major application is for eliminating casting defects. Therefore, the optimal HIP treatment could enhance the solution and precipitation of alloy, which leads to increase the mechanical properties of materials. As shown, it can increase the tensile strength of 8.8% at 25°C (854.51 MPa → 929.31 MPa), 13.0% at 540°C (831.45 MPa → 939.45 MPa), and 13.6% at 650°C (898.08 MPa → 1020.29 MPa). The 0.2% yield strength was increased by 12.8% at 25°C

(803.59 MPa → 906.2 MPa), 11.4% at 540°C (824.21 MPa → 918.38 MPa), and 13.5% at 650°C (883.89 MPa → 1003.18 MPa). Elongation also increased about 32.9% at 25°C (1.67% → 2.22%), 21.6% at 540°C (1.94% → 2.36%), and 99.5% at 650°C (1.95% → 3.89%) after tension tests. Particularly, the elongation was increased to 99.5% after 650°C tension test, suggesting that the HIP-ed specimen has the better plastic deformation resistance during high temperature conditions. The experimental results proved that the 713LC alloy has a better material strength and ductility under higher temperature, which explains why the 713LC alloy is better suited for a long time high-temperature operating conditions than other nickel-based superalloys.

Similar test results were obtained when tensile test was conducted at a very slow strain rate (0.0001 s⁻¹), as shown in Table 4, due to γ' precipitations of uniform distribution in the γ matrix after HIP treatment. Meanwhile, the uniform precipitation of γ' phase increases the mechanical strength of 713LC superalloys; most the precipitates can be considered as Ni₃Al in this study. The strengthening phases of 713LC were effectively increased *via* HIP treatment. Therefore, HIP treatment enhanced the tensile strength by 12.2% at 25°C (824.94 MPa → 925.42 MPa), and 10.1% at 540°C (878.02 MPa → 967.02 MPa). The 0.2% yield strength also increased to about 15.5% at 25°C (775.81 MPa → 895.95 MPa), and 9.6% at 540°C (853.08 MPa → 934.55 MPa). Moreover, elongation increased for 30.4% (1.81% → 2.36%) at 25°C, and 23.6% (2.25% → 2.78%) at 540°C. The experimental results showed that the tensile properties of 713LC superalloys are obviously increased and improved through HIP treatment.

Figure 3 shows the TEM micrographs and EDS result of γ' precipitation observed for the 1180°C, 175 MPa, 2 h HIP treated on Inconel 713LC superalloy. Figure 3(a) shows the bright field, and Fig. 3(b) shows the dark field of the γ' precipitates observation. The dark field of TEM photos can

Fig. 3 TEM micrographs of γ' precipitation observed for the 1180°C, 175 MPa, 2 h HIP treated on Inconel 713LC superalloy: (a) bright field, (b) dark field, EDS results for (c) matrix, (d) γ' precipitation.

be seen on the γ base with a very uniform distribution of γ' precipitations. Moreover, there is some MC carbide precipitation and distribute in the matrix. The most dramatic and effective strengthening mechanism is the precipitation of an intermetallic phase from the solid solution. Superalloys consist of the austenitic matrix phase gamma (γ) plus a variety of secondary phase. The principle secondary phases are the carbides MC, $M_{23}C_6$, M_6C and M_7C_3 in all superalloy types and prime fcc ordered $Ni_3(Al, Ti)$ in Ni-based superalloys.⁴⁾ Precipitation hardening by MC carbides in the vicinity of grain boundaries plays an important factor in improving the mechanical properties of 713LC. Enhanced tensile properties can be related to the increasing MC carbide precipitation that results from high temperature plastic deformation during tensile testing. In addition, the significantly increased elongation of specimens in tensile tests at 650°C compared with room temperature (as shown in Table 3) can be ascribed to high temperature precipitation strengthening and superplasticity effects of MC carbides in the 713LC superalloy by HIP treatment.

Relative to superalloy, formation of the γ' phase Ni_3Al in 713LC superalloy is the most important. Figures 3(c) and 3(d) show further comparison with EDS analysis of the matrix and γ' precipitates. The main alloying elements are

Ni, Nb, Cr, Ti, and Al, and the chromium content of EDS analysis is higher in the matrix. It should be the γ phase for matrix, however, the matrix precipitates of the EDS results showed that low chromium and aluminium content contrast with at a high level, thus, they should be the standard of γ' phase Ni_3Al precipitates. Significantly, the Nb content was lower, and the Al content higher, indicating Ni_3Al precipitation [Fig. 3(d)]. Therefore, it is reasonable to suggest that the major of γ' phase is Ni_3Al precipitation in this study.

Figure 4(a) shows the TEM micrographs of matrix, grain boundary, and γ' precipitation observed for the 1180°C, 175 MPa, 2 h HIP treated on Inconel 713LC superalloy. The most common phenomenon is that the γ' precipitates out first from γ matrix, and has a cube-to-cube orientation relationship with the matrix. Numerous fine γ' precipitates were observed the matrix. Extensive γ' precipitations have occurred in the γ matrix, which enhances the effect of precipitation strengthening. A previous study identified¹⁷⁾ dislocation rich slabs in the form of thin bands and ladder-like bands running parallel to $\{111\}$ planes that passed through both γ channels and ordered γ' precipitates as also contributing to creep resistance and strengthening mechanism. In this study, by using the select diffraction of γ matrix, the SAD pattern is (112) that should be the FCC structure, as shown in Fig. 4(b). Furthermore, the

Fig. 4 TEM micrographs of γ' precipitation observed for the 1180°C, 175 MPa, 2 h HIP treated on Inconel 713LC superalloy (a) dark field, (b) SAD pattern for matrix (112), (c) SAD pattern for γ' (110).

SAD pattern is (110) for γ' , indicating the same FCC structure, as shown in Fig. 4(c).

Figure 5 shows the fractographs of as-heat treated and 1180°C, 175 MPa, 2 h HIP treatment for Inconel 713LC superalloy after 25°C with different strain rates of tensile tests. Comparison of the cross-section of observation at different strain rates suggested that the as-heat treated 713LC superalloy shows the most brittle fracture after 25°C tensile testing, as shown in Figs. 5(a) and 5(c). The photos illustrate the brittle fracture microstructure after TRS tests which show the brittle intergranular failure mode.^{18,19} However, the optimal HIP treated 713LC superalloy shows the ductile fracture. Beside, Fig. 5(d) shows the very fine dimpled rupture in the interior of the grains at very slowly strain rate. According to the previous studies indicated that should be a ductile transgranular failure mode.^{18,19} Similarly, compared with as-heat treated and optimal HIP treated Inconel 713LC superalloy after 540°C with different strain rates of tensile tests. Both Figs. 6(a) and 6(c) show the crack phenomenon of obvious brittle fracture. The optimal HIP treated on 713LC superalloy shows better toughness fractures after 540°C with different strain rate of tensile tests. The cross-section of the

fractographs appears an obviously dimpled fracture, as shown in Figs. 6(b) and 6(d).

Further comparison of the fractographs of as-heat treated and 1180°C, 175 MPa, 2 h HIP treatment for Inconel 713LC superalloy after 650°C high temperature tensile tests, is shown in Figs. 7(a) and 7(b). The un-treated 713LC superalloy castings both have a brittle and ductile rupture appearance, as shown in Fig. 7(a). On the contrary, the optimal HIP treated on 713LC shows a fine dimple fracture, as shown in Fig. 7(b). Each dimple is one of a micro void that formed and then separated during the fracture process. Due to the limitation imposed by the solution and aging treatment of the 713LC superalloy, the coarse grain, porosity, and more defects, and casting process of segregation is easily resulted in intergranular fracture, both at high temperature and room temperature. Therefore, there would be reduced material damage toughness; on the other hand, the fine and uniform microstructure of 713LC superalloy appeared after HIP treatment. Furthermore, the porosity and segregation defects were improved by HIP treatment. It increased the mechanical strength and microstructure, and showed the better transgranular fracture.

Fig. 5 Fractographs of as-heat treated and after 1180°C, 175 MPa, 2 h HIP treatment for Inconel 713LC superalloy tensile specimens at 25°C (a) as-heat treated (strain rate = 0.001 s^{-1}), (b) HIP treated (strain rate = 0.001 s^{-1}), (c) as-heat treated (strain rate = 0.0001 s^{-1}), (d) HIP treated (strain rate = 0.0001 s^{-1}).

Fig. 6 Fractographs of as-heat treated and after 1180°C, 175 MPa, 2 h HIP treatment for Inconel 713LC superalloy tensile specimens at 540°C (a) as-heat treated (strain rate = 0.001 s^{-1}), (b) HIP treated (strain rate = 0.001 s^{-1}), (c) as-heat treated (strain rate = 0.0001 s^{-1}), (d) HIP treated (strain rate = 0.0001 s^{-1}).

Fig. 7 Fractographs of as-heat treated and after 1180°C, 175 MPa, 2 h HIP treatment for Inconel 713LC superalloy tensile specimens at 650°C (a) as-heat treated (strain rate = 0.001 s⁻¹), (b) HIP treated (strain rate = 0.001 s⁻¹).

4. Conclusions

In this study, the fine and uniform γ' precipitations of 713LC superalloy appeared in the γ matrix after HIP treatment. The main strengthening mechanism observed in the present specimens was coherent precipitation of the inter-metallic Ni₃Al phase. In addition, precipitation hardening by MC carbides in the vicinity of grain boundaries plays an important factor in improving the mechanical properties of 713LC. Furthermore, the porosity and mechanical properties were improved by HIP treatment. The hardness of 713LC superalloy increased to HRC 40.5; the porosity decreased by 60.3%, and the density enhanced to 7.92×10^3 kg/m³ after HIP treatment. The tension test results proved that HIP treatment can effectively increase the tensile strength, 0.2% yield strength, and elongation of 713LC alloy. Moreover, most HIP-ed specimens showed better ductile and fine dimple fractures.

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REFERENCES

- 1) M. Koizumi (ed.): Hot Isostatic Pressing: Theory and Applications, Proc. Third International Conference, (Elsevier Applied Science, 1992) pp. 135–187.
- 2) T. Garvare: Proc. Int. Conf. on Hot Isostatic Pressing-Lulea/15–17 June, (The Swedish Institute of Production Engineering Research, 1987) pp. 41–152.
- 3) Z. Jonsta, P. Jonsta, V. Vodarek and K. Mazanec: *Acta Metall. Slovaca* **13** (2007) 546–553.
- 4) M. J. Donachie: *Superalloys — A Technical Guide*, (ASM Int., 1988) pp. 15–124.
- 5) S. S. Manson: *Nonferrous alloys 713LC*, code 4108, (Aerospace Structure Metal Handbook, 1976) pp. 1–22.
- 6) F. A. Souza, F. M. Bastos and P. R. M. Jorge: *Thermal Eng.* **3** (2004) 115–121.
- 7) Z. Jonsta, P. Jonsta, J. Sojka and V. Vodarek: *Acta Metall. Slovaca* **12** (2006) 436–442.
- 8) I. S. Kim, B. G. Choi, S. M. Seo and C. Yo: *Met. Mater. Int.* **12** (2006) 1–5.
- 9) T. Podrabsky, Z. Jonsta, K. Hrbacek and K. Nemec: *Acta Metall. Slovaca* **9** (2003) 198–202.
- 10) R. R. Bowman and S. D. Antolovich: *Metall. Trans. A* **17A** (1986) 173–180.
- 11) S. H. Chang: *J. Alloy. Compd.* **486** (2009) 716–721.
- 12) S. H. Chang, S. C. Lee and K. T. Huang: *Mater. Trans.* **51** (2010) 1683–1688.
- 13) S. H. Chang: *Mater. Trans.* **50** (2009) 909–916.
- 14) S. H. Chang, K. T. Huang and C. M. Liu: *IJCMR* **24** (2011) 113–117.
- 15) Flow Autoclave System, Inc.: Hot Isostatic Pressing Applications, (1996).
- 16) T. Podrabsky, M. Julis, S. Pospisilova, K. Nemec, K. Hrbacek, A. Rek and Z. Jonsta: *Acta Metall. Slovaca* **11** (2005) 62–67.
- 17) M. Petrenec, K. Obrtlík and J. Polak: *Mater. Sci. Eng. A* **400–401** (2005) 485–488.
- 18) G. A. Rao, M. Kumar, M. Srinivas and D. S. Sarma: *Mater. Sci. Eng. A* **355** (2003) 114–125.
- 19) G. A. Rao, M. Srinivas and D. S. Sarma: *Mater. Sci. Eng. A* **383** (2004) 201–212.