	ソン　ジョンヨン
氏　　　　　　　名	宋　　全　　永
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指　　導　　教　　員	東北大学教授　千葉　晶彦
論 文 審 査 委 員	主査　東北大学教授　千葉 晶彦　東北大学教授　吉見 亨祐 東北大学教授　及川 勝成　教授　小泉 雄一郎

論　文　内　容　要　旨

Chapter 1. Introduction

Nickel-based superalloys used for turbine engine, turbine wheel, disc application show excellent room and elevated temperature mechanical properties such as creep and fatigue resistance. Their excellent mechanical properties were originated from presence of high volume fraction (close to 50%) of γ' precipitate coherently embedded within the γ phase. However, high volume fraction of γ' precipitate results in poor ductility at the room and elevated temperature. Therefore, it is very important to understand and to control the γ' precipitate.

In the last three decades, many researcher have been attempted to improve the ductility of nickel-based superalloy through heat treatment and modification of γ' precipitate. Especially, Nathal et al. [1] found that Co addition on the nickel-based superalloy results in increase in tensile ductility. Co addition reduces the γ' solvus temperature as well as MC carbide solvus temperature. These microstructural changes can affect the mechanical properties such as room temperature ductility.

Chapter 2. Microstructure and mechanical properties of IN-713C alloy before and after solution heat treatment

IN-713C alloy used for turbine wheel of turbocharger is generally produced by investment cast because this alloy shows low ductility. Investment cast is considered to be optimized cast method for IN-713C alloy. However, investment cast has low cooling rate, which results in irregular shape γ' precipitate and coarse MC type carbide. These microstructural disadvantages give rise to degradation of mechanical properties such as yield, ultimate strength and elongation. Metal mold cast is used to produce the homogeneous microstructure without irregular shape γ' precipitate and coarse MC type carbide. Fig. 1 shows microstructures before and after solution heat treatment. As shown in this microstructures, metal mold cast IN-713C alloys before and after solution heat treatment show much smaller γ'- precipitate than those of investment cast IN-713C alloys before and after solution heat treatment. Also, metal mold cast IN-713C alloys do not exhibit coarse MC carbides. Fracture mode is a mixed transgranular ductile fracture and transgranular cleavage fracture. The fracture of these IC/MMC alloys before and after solution heat treatment was originated from interface between γ phase and γ' precipitate, and coarse MC carbides. This is due to high cooling rate of metal mold cast. High cooling rate suppresses the nucleation of γ' precipitate, and results in homogeneous chemical distribution of γ' forming element such as Al. The absence of irregular shape γ' precipitate and coarse MC carbide results in increase in tensile ductility. Metal mold cast IN-713C alloy shows excellent tensile strength and elongation compared to investment cast IN-713C alloy.

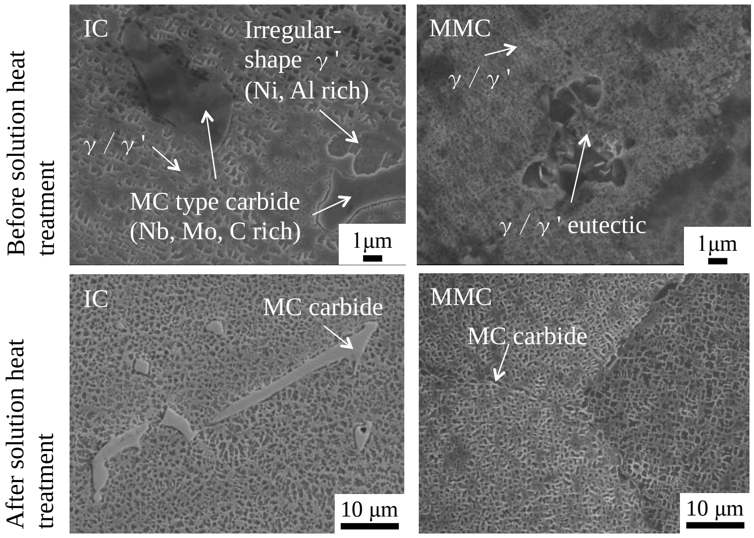

Figure 1. Microstructures of IN-713C alloys before and after solution heat treatment.

Chapter 3. Effect of cobalt addition on the microstructure and mechanical properties after solution heat treatment

We prepared three kinds of IN-713C alloys with different Co content, 0 mass% Co (0Co), 5 mass% Co (5Co), and 10 mass% Co (10Co). It is well known that Co addition produces the decrease in γ' solvus temperature, which results in decrease in γ' precipitate size. Fig. 2 shows FE-SEM micrographs of IN-713C alloys after solution heat treatment at 1473 K for 2 h and subsequently furnace cooled. 5Co and 10Co alloys show split behavior. However, 0Co alloy does not exhibit split behavior. This is due to the presence of misfit dislocation. The split behavior of γ' precipitate is dependent on elastic energy. 0Co alloy containing misfit dislocation produces relaxation of elastic energy, which results in γ' precipitate growth without split behavior. The decreasing γ' precipitate size produced

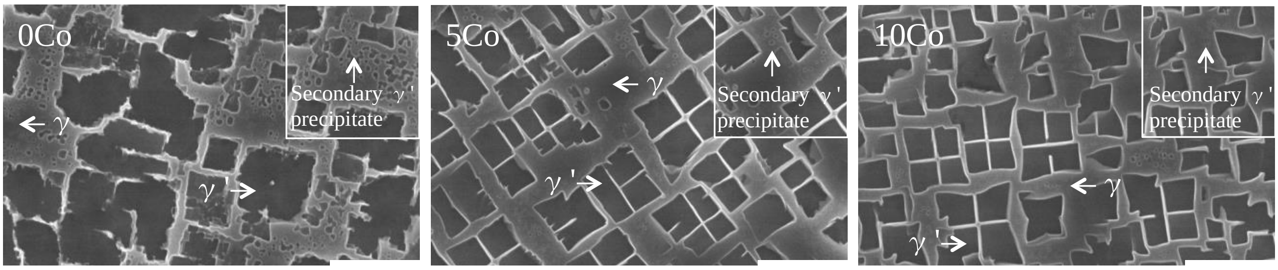

Figure 2. FE-SEM micrograhps of IN-713C alloys after solution heat treatment at 1473 K for 2 h and subsequently furnace cooling.

the decrease in tensile strength and increase in tensile ductility. The result of room temperature tensile test shows that IN-713C alloys with and without Co addition exhibit excellent tensile elongation, and tensile elongation was increased with increasing Co addition. Generally, mechanical properties such as elongation are significantly dependent on γ' precipitate size. With decreasing γ' precipitate size, dislocation movement can be changed from bypassing of γ' precipitate to cutting of γ' precipitate. 0Co alloy has larger γ' precipitate size than that of 5Co and 10Co alloy. Therefore, cutting of γ' precipitate is more difficult compared to 5Co and 10Co. On the other hand, 5Co and 10Co alloy have relatively smaller γ' precipitate size. Thus, cutting of γ' precipitate is easy. Also, we can consider the anti-phase boundary (APB) produced by cutting of γ' precipitate. If pair of dislocations encounter widely dispersed γ' precipitates, leading dislocation is interrupted. The leading dislocation produced APB, which can be annihilated by subsequent dislocation. Fracture mode is similar to cast IN-713C alloy. Fracture surfaces are comprised of large amount of dimples. The large amount of dimples were originated from interface between γ phase and γ' precipitate. It suggests that fracture mode is transgranular ductile fracture. Totally, IN-713C alloys with and without Co addition show excellent room temperature tensile properties. With increasing Co addition, room temperature tensile ductility increases through decrease in γ' precipitate size.

Chapter 4. Cold press and recrystallization behavior

This chapter is concerned with annealing behavior of IN-713C alloy with and without Co addition. After solution heat treatment, IN-713C alloys were subjected to cold press to the final thickness reduction of 30 %. Cold pressed samples were isothermally annealed for various times at 1373 K to 1473 K (step: 50 K) and subsequently oil quenched. After isothermal annealing heat treatment, deformed microstructures are replaced by a microstructure with low dislocation density. Fig. 3 shows temporal change in hardness (Fig. 3(a)) and recrystallized fraction (Fig. 3(b)). When isothermal annealing temperature is 1473 K, the hardness values of alloys drop rapidly in short time. However, when annealing temperatures are 1373 K and 1423 K, hardness values drop so slowly taking long time. The results of hardness test suggest that, softening process is significantly dependent on isothermal annealing temperature. Transition from deformed microstructure to fully recrystallized microstructure occurs rapidly with annealing treatment at 1473 K. This annealing temperature is above γ' precipitate solvus temperature. However, when annealing temperature is 1423 K, and 1373 K, which are above /almost same as the γ ' solvus temperature, the existence of γ' precipitates interfere the recrystallization. Fig. 4 shows the microstructures of IN-713C alloys subjected to isothermal annealing treatment at 1373 K for 55.5 hours and subsequently oil quenching. All of the alloys show inhomogeneous grain size distribution, and show discontinuous γ' precipitations. 0Co alloy shows irregular shaped discontinuous γ' precipitation. The size of discontinuous γ' was decreased with increasing Co content. It is due to increase in the solubility of γ' achieved by Co content. Co addition increases the solubility of γ' precipitate into γ matrix. In consequence, recrystallization rate can be increased with increasing Co content.

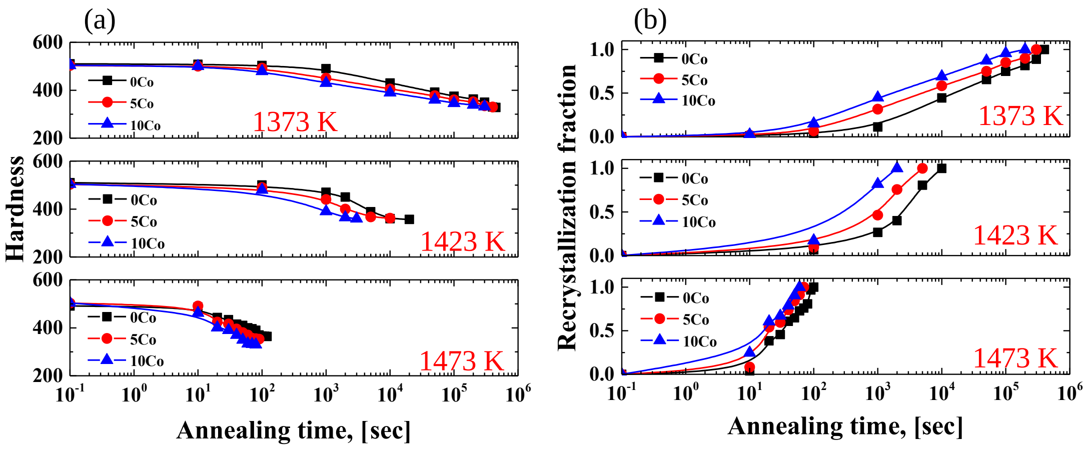

Figure 3. Temporal change in hardness and recrystallized fraction of IN-713C alloys with different Co content.

Chapter 5. Room temperature tensile behavior of recrystallized microstructure

Room temperature tensile properties such as strength and elongation of recrystallized IN-713C alloys with and without Co addition were investigated. We prepared three kinds samples with different Co content, 0 mass% Co (0Co), 5 mass % Co (5Co), and 10 mass % Co (10Co). After cold press to the final thickness reduction of 30 %, samples were subjected to isothermal annealing heat treatment at 1473 K for 100/300 sec and subsequently oil quenching. Recrystallized microstructure including γ ' precipitate is significantly dependent on Co content. After isothermal annealing heat treatment at 1473 K for 100 sec and 300 sec, 10Co alloy has a much smaller size

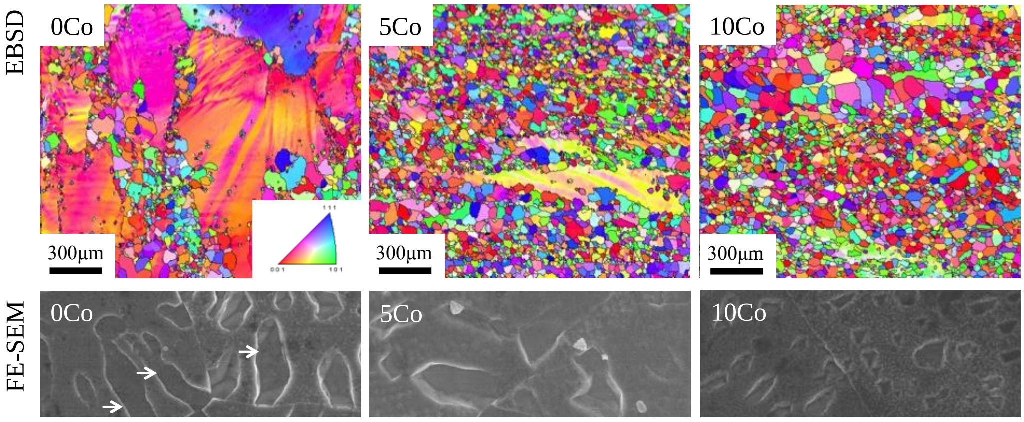

Figure 4. IPF map and FE-SEM micrographs of IN-713C alloys after isothermal annealing heat treatment for 55.5 h and subsequently oil quenching.

distribution of γ' precipitate compared to 0Co and 5Co alloys. The results of tensile test in this chapter are similar to the results of tensile in Chapter 3. All of the alloys exhibit excellent tensile ductility above 20 % tensile elongation. 10Co alloy exhibits much higher tensile ductility than that of 0Co and 5Co alloys. Especially, Considere's instability criteria, expressed by $d\sigma/d\varepsilon < \sigma$, can be seen in IN-713C alloys annealed for 300 sec.

Chapter 6. Conclusions

Microstructural evolution, recrystallization, and mechanical properties such as strength and ductility have been investigated using IN-713C alloy with and without Co content. Generally, IN-713C alloy is well known to exhibit low ductility. However, present study shows that IN-713C alloy is not brittle material. And Co content give rise to increase in tensile properties through modification of γ' precipitate. IN-713C alloys with Co content exhibit a much higher tensile ductility than that of IN-713C alloy without Co content maintaining high strength.

Reference

[1] M. V. Nathal et al. The influence of cobalt on the microstructure of the nickel-base superalloy MAR-M247, Metall. Trans. 13A, 1982, 1775-1783

論文審査結果の要旨

本論文は、タービンブレード用ニッケル基超合金 IN713C の組織制御と延性改善を目指した組織制御ならびに Co 添加の影響を調べ、本合金の延性向上を達成した一連の研究成果をまとめたものであり、以下の６章で構成される。

第１章では、研究の背景、目的、論文の構成を述べている。

第２章では、IN713C 合金の精密鋳造材と金型鋳造材の組織と室温の機械的特性についての研究成果を述べている。精密鋳造は同合金の製造に現在広く使用されている鋳造法である。しかし、精密鋳造された IN713C 合金は高強度を有すものの延性が低く、その向上が求められている。本合金の優れた機械的特性はγ'析出物の大きさ、分布、形状に支配されるため、γ'析出物の制御が重要である。精密鋳造における冷却速度は遅く、粗大な樹枝状晶、γ'析出物、炭化物が形成され、これらの粗大な組織、中でも粗大なγ'析出物と炭化物はこの合金の機械的特性、特に延性を低下さる。一方、金型鋳造は、精密鋳造より冷却速度が高く、均一微細な樹枝状晶、γ'析出物、炭化物を形成するため、室温強度と延性を向上させる。これは溶体化・時効熱処理後の組織にも影響し、金型鋳造材では、γ'析出物がより均一に分散した均質な組織を形成するため、精密鋳造材よりも高い延性を有すことを明らかにしている。

第３章では、IN713C 合金の延性に対する Co 添加と無添加の影響について述べている。さらに延性の向上を目指し、て、第２章に記した金型鋳造材を用いて、IN713C 合金への Co 添加の影響を熱処理後の組織観察ならびに力学試験により評価している。Co 添加はγ'析出物の solvus 温度を下げるためγ'析出物の大きさ、分布、形状に影響するとの考察に基づき、本章では IN713C 合金への 5%Co 添加ならびに 10%Co 添加した際の、γ'析出物の変化とその延性への影響を述べている。γ'析出物の大きさと形状は Co 添加と密接な関係があり、Co 量が増加するほど均一微細なγ'析出物を得ることができる。γ'析出物は転位の障害物なり、転位はγ'析出物のまわりに転位ループを残して通過するか、γ'析出物をせん断して通過することになる。γ'析出物が小さいと転位はγ'析出物をせん断して通過する。5%Co 添加ならびに 10%Co 添加した IN713C 合金の場合、γ'析出物が小さいため転位の動きが易くなり、延性を向上させたとしている。そして、Co 添加はγ'析出物の成長機構に影響を与えることも示している。Co 無添加 IN713C 合金ではミスフィット転位が観察されるのに対し、Co 添加 IN713C 合金ではミスフィット転位が観察されないことを示した。ミスフィット転位が存在する場合は弾性エネルギーが緩和されるためγ'析出物は分裂せずに成長する。ミスフィット転位が存在しない場合は弾性エネルギーが緩和されないため、分裂しながら成長することになる。このような Co 添加のγ'析出物の大きさ、分布、形状に対する影響が延性を向上させると結論づけている。

第４章では冷間加工した IN713C 合金の再結晶における Co 添加の影響について述べている。熱処理した IN713C 合金を室温で冷間プレスして、焼鈍の後急冷して再結晶組織を得ている。Co 添加によって均一微細なγ'析出物を得られることが第３章で明らかにされたことを踏まえて、再結晶後のγ'析出物への影響を調べる実験と考察を加えている。Co 添加した IN713C 合金は無添加の IN713C 合金より再結晶速度が増すことを示し、これをγ'析出物の大きさと関係づけている。γ'析出物は再結晶時に粒界の障害物として作用するため再結晶速度を低下させる。γ'析出物が小さいほど障害物としての機能が軽減し再結晶速度が上昇する。その結果、Co 添加によって再結晶の速度が速くなると結論づけている。

第５章では再結晶した IN713C 合金の延性への Co 添加の影響について述べている。基本的変形機構は無加工材のものと比べて変わらないものの、Co 無添加合金より Co 添加の合金が優れた延性を有すことを示している。

第６章では本研究の総括を述べている。本研究から得られた重要な知見として、金型鋳造により組織を均一にすることで延性が向上し、再結晶による組織制御も可能となり、さらに Co 添加によって組織、特にγ'析出物を微細にすることで、室温延性が改善されることを挙げている。これらの知見は、工学の発展に大きく寄与するものである。

よって、本論文は博士（工学）の学位論文として合格と認める。